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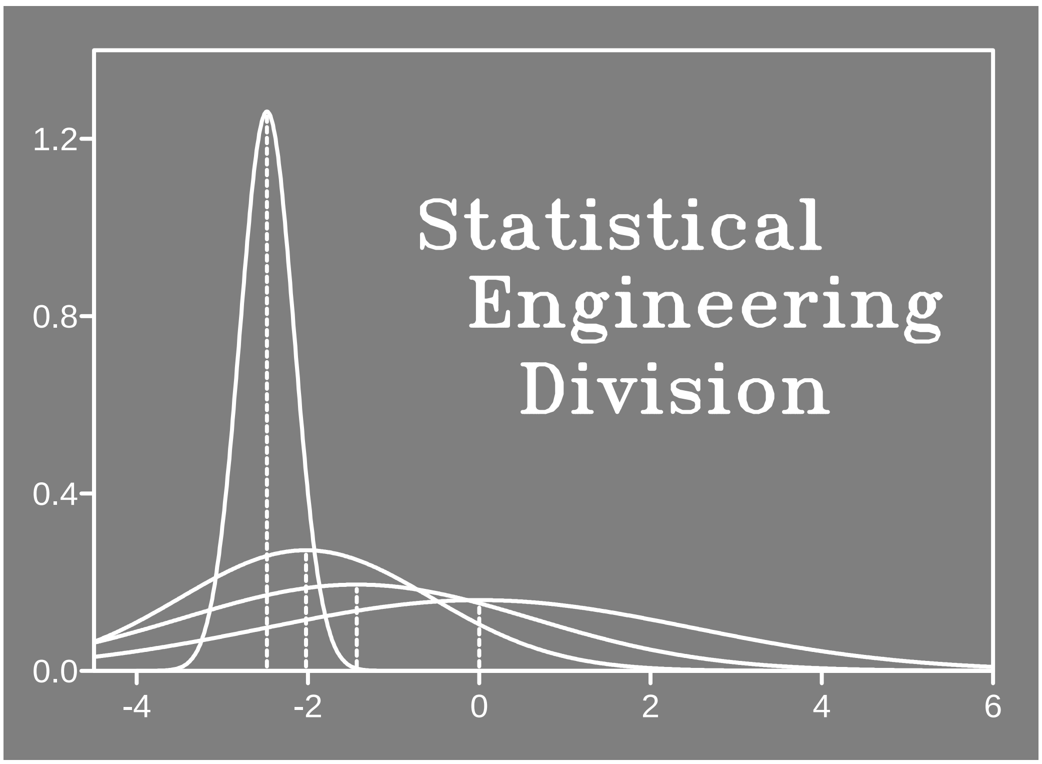
Report of Activities:

Statistical Engineering Division



U. S. Department of Commerce
National Institute of Standards and Technology
Information Technology Laboratory
Gaithersburg, MD 20899 USA

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REPORT OF ACTIVITIES IN THE STATISTICAL ENGINEERING DIVISION

MARCH 1997

Covering Period: January 1996 – March 1997
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Contents

1. Division Overview

Lynne B. Hare, Chief
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The Statistical Engineering Division (SED) is a unit of the Information Technology Laboratory of the National Institute of Standards and Technology (NIST). SED collaborates in NIST measurement science and technology research programs to support US industry through design of experiments, statistical modeling, and analysis and interpretation of data. We participate in the Laboratory's interdisciplinary research and development teams to advance information technology; we contribute to the development of appropriate statistical methodology, building on a foundation of pertinent topics in probability and mathematical statistics; and we provide leadership and computational tools to facilitate the implementation of modern statistical design, analysis and process control procedures.

The Division operates from both the Gaithersburg, Maryland, and the Boulder, Colorado, campuses of NIST with professional staff composed of Ph.D. and Masters degreed mathematical statisticians, of whom 16 are assigned to the Gaithersburg site and 4 are assigned to Boulder. This full-time staff is augmented by several faculty appointees, guest researchers and post doctoral students. A staff listing appears in Section 2.

Divisional priorities are driven by the need to support the NIST mission in the areas of:

- Promoting improved use of information technology through the NIST laboratories and outreach to industrial partners,
- Engaging in fundamental research in measurement sciences,
- Facilitating the Calibration and Standard Reference Materials programs, and
- Collaborating in high visibility projects of national interest.

This report provides technical summaries of some key project activities from January, 1996 to March, 1997 and a compilation of staff activities during that time. The project summaries are intended to provide a sampling or representative overview, not a representative summary of all Division activities. It is important that readers understand that all of this work is done collaboratively with other scientists and engineers, not by statisticians alone.

Indeed, there are many activities of SED that cannot be represented here. Additional information can be found on the SED World Wide Web Home Page by accessing the following URL: http://www.nist.gov/itl/div898/.

Thank you for reading. We welcome your comments. Please address them to:

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3. Project Summaries

3.1. Promulgation of Measurement Standards

3.1.1. Resistivity Standard Reference Materials

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The purpose of this project is to produce seven issues of SRMs with certified resistivities at 200, 100, 25, 10, 1, 0.1 and 0.01 ohm.cm levels. The SRMs come from crystals that have been grown from liquid silicon doped with phosphorous.

The SRMs are intended for on-line calibration of instruments used in semiconductor fabrication. The project began several years ago with the validation of a more precise method (relative to the existing ASTM standard method) for measuring resistivity (or sheet resistance) of silicon wafers with probing instruments.

This is a classic situation of a unit of measurement that is defined solely by a measurement method (instrumention and procedures) together with certified artifacts disseminated by a national laboratory. The research phase for this project studied failure modes, wafer stability, instrument geometries, effects of wear on probes, effects of repeated probing on wafer surfaces, and photo-electric effects (exposure to light). These experiments uncovered some mechanisms which are not yet fully understood, although physical arguments can be made by way of explanation, and suggested sources of uncertainty to be examined during the certification process.

For the certifications, the same experimental design was applied to all seven issues. A check wafer, chosen at random from the batch of approximately 150 SRMs from a single crystal, was measured daily to estimate components of variance. A pre-certification experiment on five wafers was conducted to: identify the probe with the best precision, test the difference between two wiring configuations for that probe, estimate systematic differences among the five NIST probes, and estimate temporal components of variance. This phase was followed by the certification where all wafers from the crystal were measured with a single probe. This, in turn, was followed by a post- certification experiment, identical to the pre-certification experiment, that checked for drift in the process.

Sources of type A uncertainties are: 1) probe precision; 2) run-to-run variability; 3) long-term variability in the measurement process; 4) bias in the certification probe; and 5) differences between wiring configurations for a single probe.

Type B uncertainties arise from uncertainties in the calibrations associated with the measurements of ratio of current to voltage, temperature, and wafer thickness. Problems encountered in the analyses include the effect of debris on the surface of some wafers from repeated probing and significant photo-electric effects.

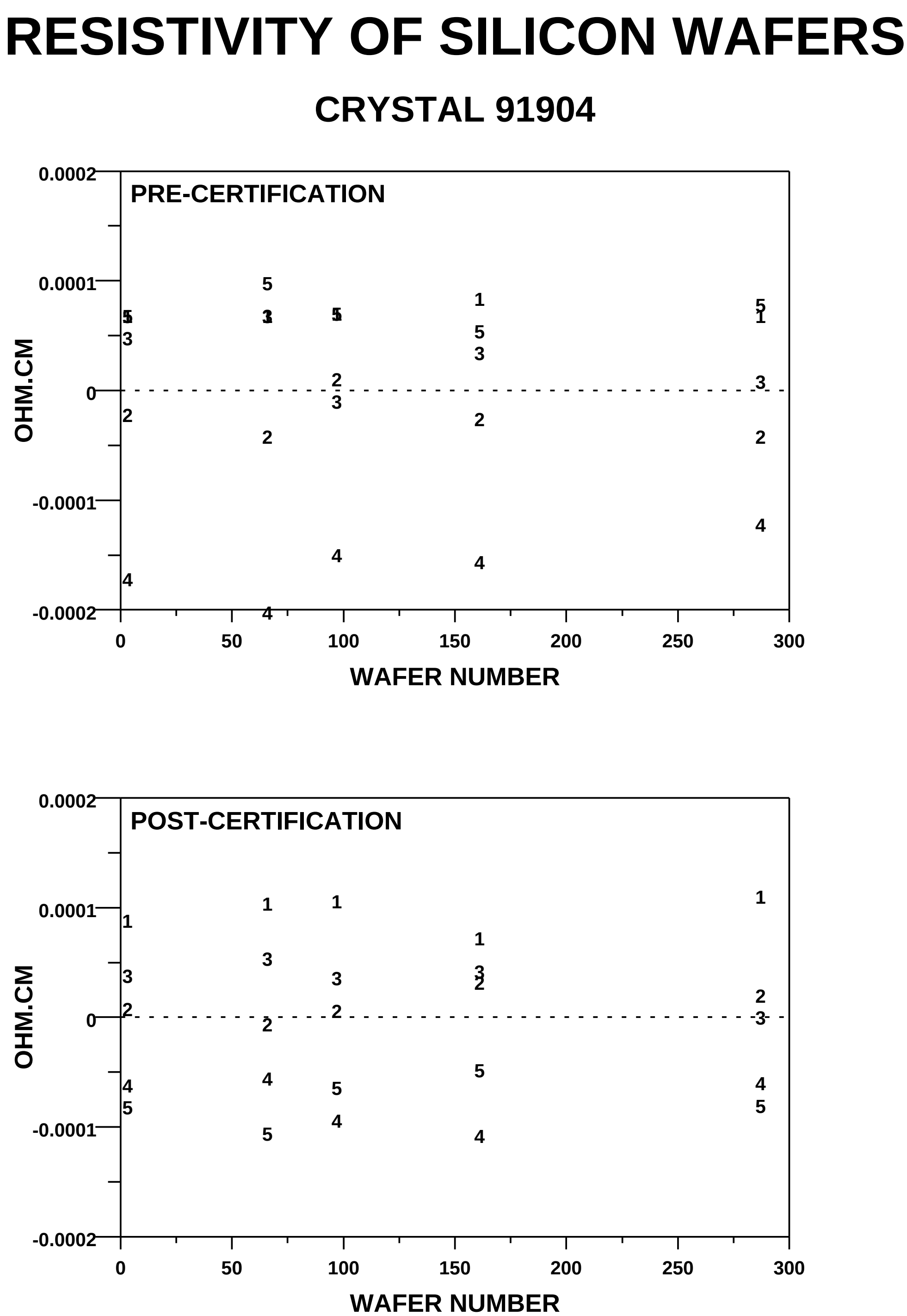

Figure 1: Differences from the wafer mean (ohm.cm) for each of 5 wafers show systematic differences among the 5 NIST probes coded (1-5).

3.1.2. Certification of Glass Beads - Particle Size Distribution, SRM 1018b

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This Standard Reference Material is intended primarily for use in evaluating and calibrating particle size measurement instrumentation covering 220 microns to 750 microns range. The SRM consists of a single bottle containing approximately 87 g of solid spherical soda-lime glass beads. Typical use is in the evaluation of wire-cloth test sieves.

The certified cumulative volume (mass) distribution was determined using both calibrated scanning electron microscopy (SEM) and standard sieving procedures on samples chosen using a stratified random sampling selection plan. The sieve analyses of ten bottles were used to determine the heterogeneity properties of the material, as well as for a comparison with the SEM results. The sieving process, which is similar to how the SRM will be employed by the customer, was used to assess the material heterogeneity. An estimate of the material heterogeneity was estimated for each sieve. Since the variability was similar for each of the sieves, a pooled estimate of material variability was included in the overall uncertainty.

The certified values were determined from the average of results from SEM analyses on five bottles. Several hundred particles were measured by SEM for each sieve fraction for a total of approximately 3000 beads measured per bottle. Particle size distributions describing the percentage of mass represented by beads with diameters in increments of 5 microns were calculated.

The certified value at each diameter is a mean percentile. The stated uncertainty is based on a 95% prediction interval. It includes allowances for measurement error (assessed from the SEM data) and material variability (from the sieving experiment).

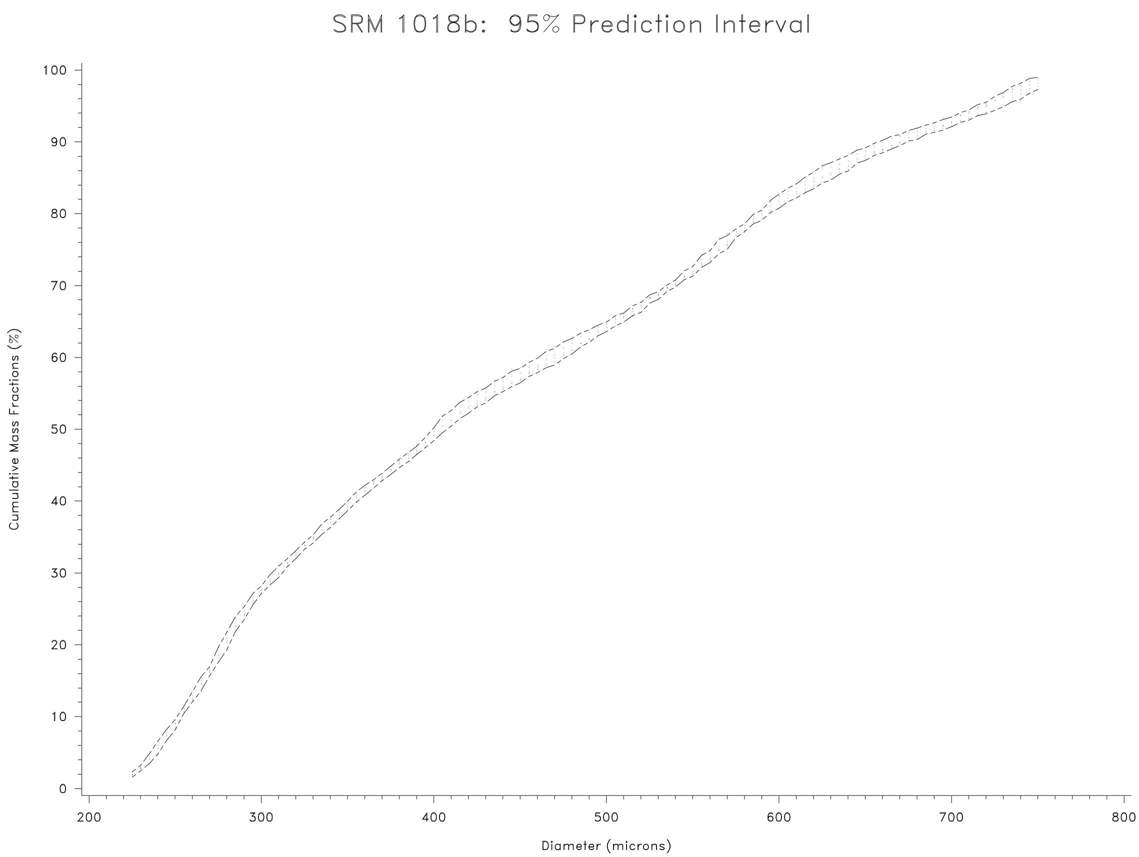

Figure 2: Cumulative distribution with 95% prediction bands

3.1.3. Test Structure Design for Electrically-Based Calibration of Optical Overlay Measurement Instruments

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Misalignment of circuitry layers on IC chips often results in defects or degraded chip performance. Thus when selecting chip manufacturing methods or monitoring output, the ability to determine the amount of misalignment (called overlay) is important.

Overlay is typically measured using high-volume optical instruments. These instruments, however, are susceptible to systematic errors called tool-induced shifts (TIS). TIS can be minimized with 'shift management techniques', but cannot be eliminated. However, development of a test structure allowing comparison of optical measurements with more accurate overlay determinations would allow reliable correction for TIS. Not coincidentally, electrically-based methods are good candidates for this application. They are relatively precise and are insensitive to many sources of error affecting optical instruments.

Simulated data and analysis results from the electrical portion of a new test structure design are shown in the accompanying figure. The data from this structure (upper left) is discrete, indicating electrical contact between vias (wires connecting circuit layers) and one of two underlying bars separated by a space. The vias have built-in offsets (o) which, with the unknown overlay (OL) and noise, control whether or not contact will be made.

To extract an estimate of OL, replicate substructures are embedded in each test structure. This allows the proportion of vias with each built-in offset that contact the first bar to be estimated (upper right, lower plot). The proportion of vias contacting the second bar is computed similarly (upper right, upper plot). The contact/noncontact transition points between the vias and each bar equal $OL \pm C$ respectively, where C is a constant related to the difference in size between the vias and the space between the bars.

The transition points are estimated by fitting generalized linear models

$$p = \frac{\exp(\beta_0 + \beta_1 o)}{1 + \exp(\beta_0 + \beta_1 o)} + \varepsilon$$

to the data by maximum likelihood with (scaled) binomial errors. These models are then linearized using the logistic transformation, $\log(p/(1-p))$, to allow computation of approximate uncertainties for the transition point estimates. Under the linearized models the transition points are given by the x-axis offset values associated with y-axis values of zero (lower left plots).

Finally the estimated transition points are averaged to estimate OL. Their uncertainties, along with a rough estimate of their correlation, are combined into an overall uncertainty which is an approximate 95% confidence interval for OL.

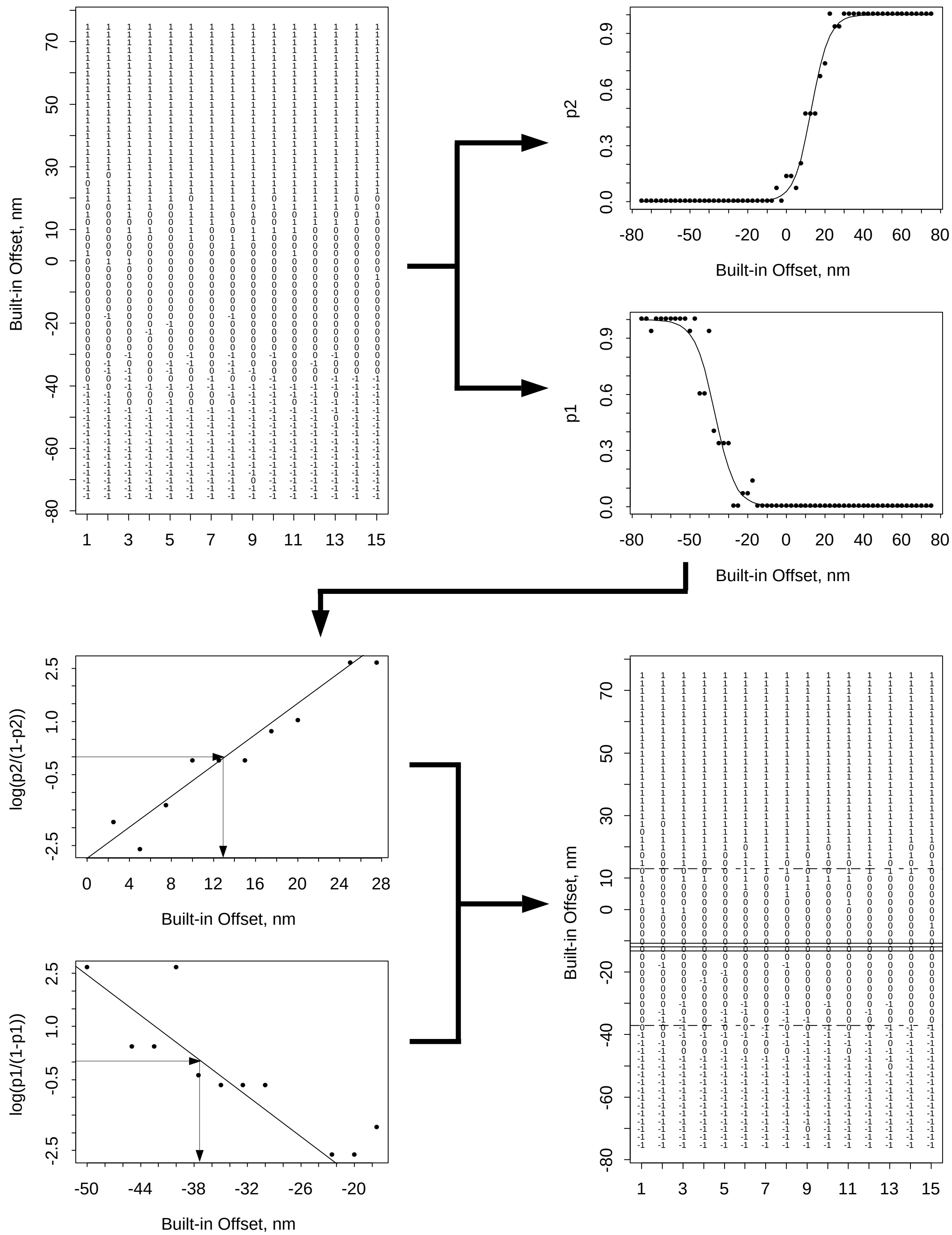

Figure 3: Simulated data from a prototype test structure with accompanying data analysis and results. The dashed lines on the final plot indicate transition points and the solid lines indicate the estimated overlay (center line) and uncertainty. The uncertainty is an approximate 95% confidence interval. The estimated overlay in this example is -12.001 ± 1.255 nm. The true overlay, from the simulation input, is -12 nm.

3.1.4. Modeling the Effect of Kelvin Voltage Taps on Electrically-Certified Linewidth Reference Materials

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As in most types of manufacturing, reference materials are used with integrated circuits (IC's) to ensure that the measurement processes used for quality control and manufacturing process characterization are accurate. Linewidth measurement procedures, which measure the width of Al 'wires' incorporated in a chip to connect circuit components, are one type of procedure commonly validated using reference materials.

The systems used to measure linewidth in production include a variety of scanning beam and scanning probe systems. Certification of reference materials can be done with accurate, laboratory versions of scanning systems, or by electrical methods. Electrical methods have the advantages of being relatively precise and insensitive to many of the sources of error affecting the other systems.

Unlike many other types of manufacturing, the reference materials used with IC's must have test structures which simulate circuit features added to the material. In the case of linewidth, test structures consist of a length of conductor called a bridge that is connected to voltage taps at each end which link it to pads where the electrical measurement instrumentation is connected.

Until recently, designs for electrically-certifiable linewidth test structures were limited by the need to keep voltage taps narrow relative to the linewidth. Wide taps effectively shorten the bridge length by slightly increasing the surface area of the conductor, increasing its measured linewidth. New test structures designed at NIST, however, eliminate the need for restrictive design rules by correcting for the effective shortening of the bridge, denoted δL. The new test structures increase the spatial resolution of the reference material measurements, but require relatively complex nonlinear analysis to determine linewidths.

To ensure that the results of linewidth data analysis from a monocrystalline Si reference material were accurate, finite element calculations of δL were made for confirmation. Because the computations are time consuming and are needed for test structures of many different linewidth sizes, development of a simple model relating δL and the linewidth was undertaken, rather than direct comparison of measured and calculated results. δL also depends on the tapwidth and a measure of the incomplete etching of the corners between the line and taps, called the facet size, so those factors were also included.

Two considerations dictated the choice of the experiment design. First, the form of the

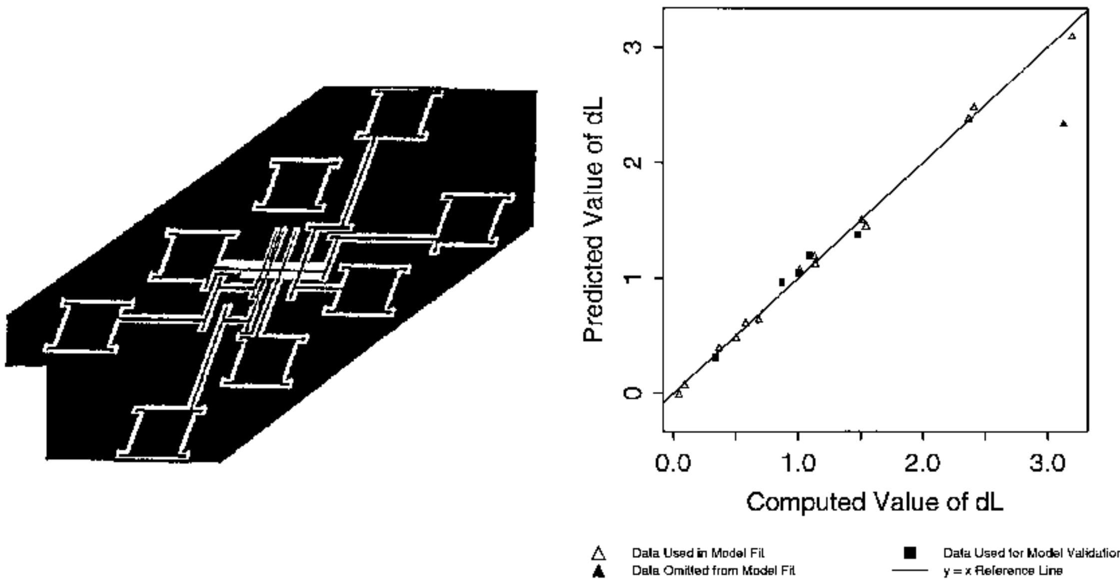

Figure 4: Linewidth test structure in monocrystalline silicon reference material (left). Predictions from the limited-scope model for δL versus finite element 'data' (right).

model was not known, but scientific knowledge suggested δL should vary smoothly and slowly with the varying inputs. The second point was the expense of the finite element calculations. As a result of these considerations, a variant of the central composite design was chosen. This design allows estimation of a full quadratic model in three factors and requires only fifteen data points.

The initial fit of the full model to the data and subsequent graphical residual analysis suggested that the model fit well except for one outlier. Omitting the outlier and refitting the model verified that it fit the remaining data well and could be simplified even further by dropping two interaction terms.

In this situation, selection of an appropriate model hinges on the interpretation of the outlier. The finite element calculations were verified to be correct, however, and extensive additional data collection was not an option. As a compromise, the outlier was omitted from the analysis and the scope of the model was limited to an appropriate region.

Because of the outlier, and the fact that the data is used primarily to estimate parameters rather than to detect lack of fit with this design, it seemed prudent to test the model with some independent data. A comparison of predictions from the model to finite element responses for five random test points is shown in the accompanying figure. From the figure it is clear the model predicts the values of δL observed at the test points well, evidence that the limited-scope model is reasonable.

The δL predictions from the model matched the experimental results reasonably well, directly confirming the benefit of the δL correction and helping verify the suitability of test structures replicated in monocrystalline Si as measurement references.

3.1.5. Computational Metrology of Manufactured Parts

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The accurate determination of the dimensions of manufactured parts is fundamental to the production of quality products. A coordinate measuring machine (CMM) offers an effective and flexible solution to the problem. U.S. industry alone uses more than 20,000 CMMs. However, there is currently no rigorous methodology to determine the accuracy of the measurements from a CMM. Consequently, CMMs are considered untraceable to the SI according to ISO 9000 definitions. Developing such traceability methodology for CMMs would (1) promote improvement in quality and efficiency through better determination of part dimensions and (2) facilitate international trade that requires ISO 9000 compliance. As part of a NIST competency project, SED scientists play an active role in a cross-disciplinary group developing traceability methods.

Basically, a CMM is a robotic machine that positions a sensing probe in its working volume. The probe contacts a sample of locations on the part surface and the CMM records corresponding three-dimensional point coordinates. The measurement process contains many sources of uncertainty. Some of the largest sources are the geometric distortions of the machine frame, the systematic effect of the probe, and thermal and mechanical effects of the operating environment. In the first two years of the project, our group developed a reliable model for real-time correction of the systematic effect of the probe. The result is an improved system without significant added costs. The paper "Error Compensation for CMM Touch Trigger Probes" by Estler et al, published in *Precision Engineering* in 1996, summarizes the results.

Currently, our group is working on the next large source of uncertainty, the geometric distortions of the machine frame. In order to fully describe these distortions, a high-dimensional ($\mathbf{R}^3 \rightarrow \mathbf{R}^6$) function is required. Such a function would require an inordinate amount of data to estimate. Based on established results, our group is attempting to account for the bulk of the distortions with a mathematical model of the rigid-body mechanics of the CMM. We are currently exploring designs to efficiently estimate these models. The accompanying figure displays 28 different positions of a calibrated artifact in the working volume of the CMM. The shaded positions are a minimum set required to estimate the model. Additional positions provide information to assess the validity of the model. Eventually the model will be used to predict the uncertainty of CMM measurements and will form part of a traceability methodology.

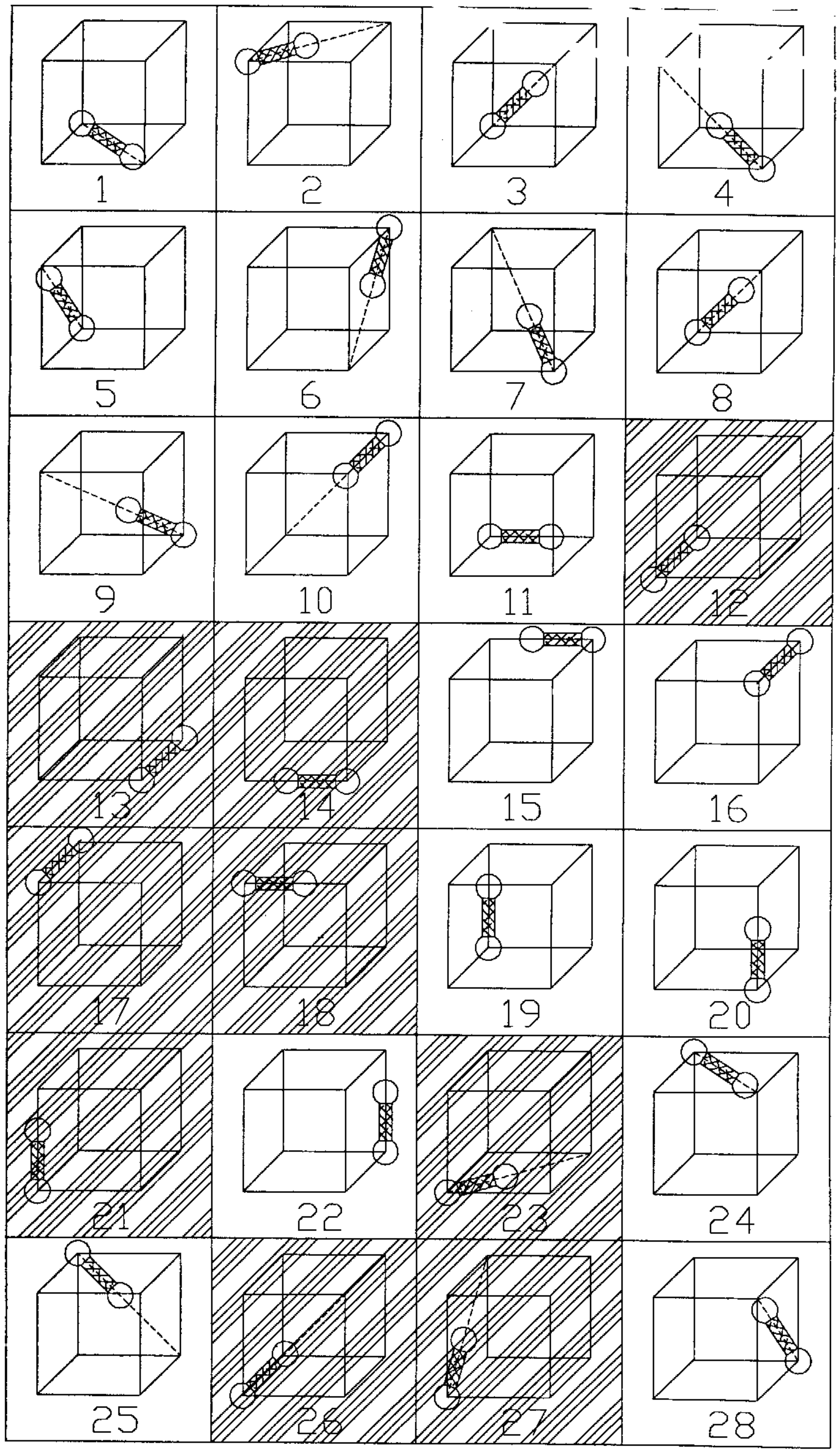

Figure 5: Calibrated artifact positions.

3.1.6. Test Blocks for Rockwell C Scale Hardness–Standard Reference Materials

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Across the surface of an HRC test block, the hardness is not uniform despite care in block manufacture. The effects of this nonuniformity on results can be incorporated into the uncertainty ascribed to the results. More can be done, however. The standard deviation of the hardness difference for two points 5 mm apart is half that of the standard deviation for large spacings. By taking this into account, one can determine differences among hardness testing machines and indentors more precisely. When the points on the block are chosen properly, the uncertainty reduction might be much more than 50 percent.

One only compares hardness measurements with each another. Sometimes this is an in-house comparison as in the use of hardness measurement to control a manufacturing process, and sometimes this is a contractual comparison to see if a supplier met customer specifications. There is no comparison with a qualitatively different measurement because hardness cannot be computed by scientific theory from any other measurement. For example, there is nothing similar to the mass balance equations that connect an assay of gold ore with the amount of gold extracted from the entire ore lot. Thus, the only issue is the relation of hardness measurements made under one set of circumstances with those made under another.

Ideally, hardness measurements to be compared will be made with exactly the same equipment and procedure, but of course, they can't be. Test blocks are used to characterize differences among the ways measurements are made. Based on this characterization, one might adjust the measurement procedures or one might determine calibration equations. In any either case, the nonuniformity of the test blocks interferes with the characterization and limits one's ability to make different measurement systems produce nearly identical results. Thus, a method for reducing the effects of the nonuniformity serves to diminish a fundamental limitation on the comparability of hardness measurements.

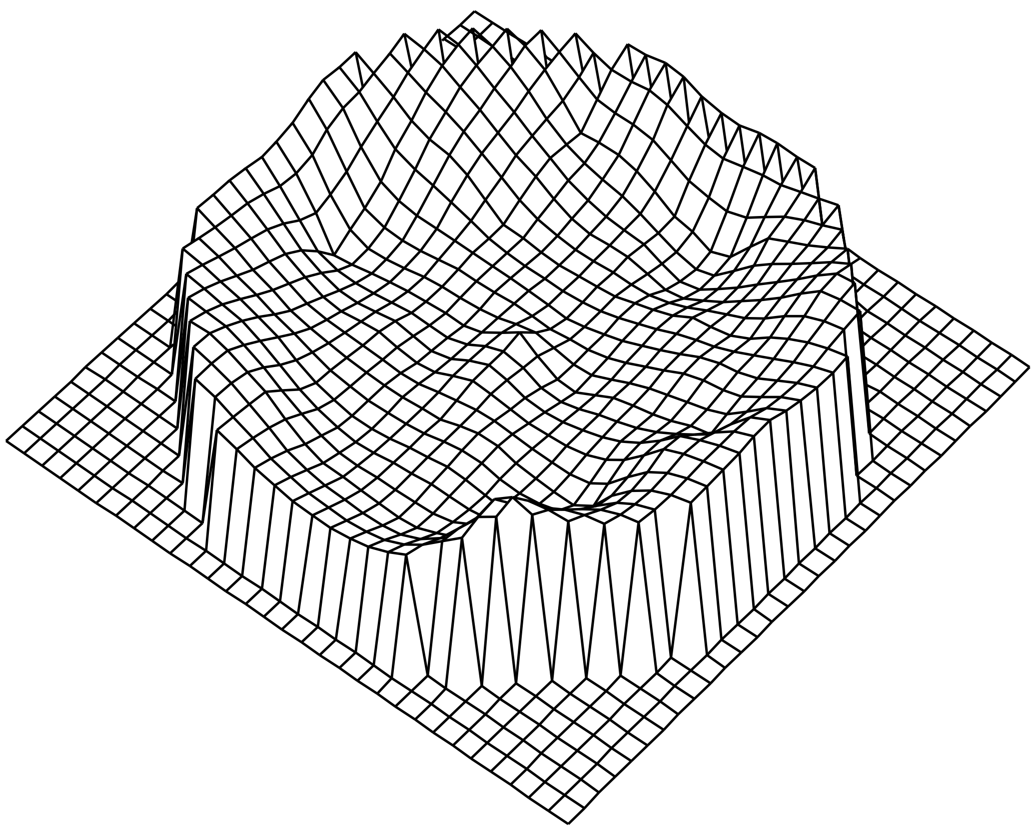

Figure 6: Hardness variation across a test block with 28 mm radius, HRC range 64.670 - 64.865.

3.1.7. Calibration of High Speed Oscilloscopes

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The design of low cost lightwave communications systems requires accurate measurements of the response of optical to electrical converters in both magnitude and phase. The frequency range of interest is about 1 MHz to 50 GHz or more. To meet this need NIST is investigating methods to calibrate the frequency response of equivalent time sampling devices (both optical and electrical) with impulse or sinusoidal stimuli. Different methods will be used to cross check these calibrations.

In this work, a high–speed sampling oscilloscope automatically can produce histograms comprising thousands of quasi–random–time samples from input waveforms swept over many frequencies and power levels. The model for N random–time samples from a signal generator under test is given by

$$V_j = a_0 + a_1 \ \sin(2\pi f t_j) + a_k \ \sin(2\pi k f t_j + \phi_k) + e_j$$

where $\{t_j, j = 1, 2, \ldots, N\}$ are independently and uniformly distributed on $[0, 1/f]$, and the e_j's denote white Gaussian noise with variance σ^2. The amplitude a_1 is the main parameter of interest, but the amplitude and phase, a_k and ϕ_k, are to be estimated if the harmonic term is detected. We have obtained the first twelve moments of the sampling distribution of V for the most likely situations of a second or third harmonic term ($k = 2$ or 3). For deriving method–of–moments estimates of the parameters, we convert from moments to cumulants, since the latter quantities are simpler expressions than the former. For instance, the first five cumulants for the second harmonic model are: $\kappa_1 = a_0$, $\kappa_2 = \sigma^2 + (a_1^2 + a_2^2)/2$, $\kappa_3 = -(3/4)a_1^2 a_2 \sin(\phi_2)$, $\kappa_4 = -(3/8)(a_1^4 + a_2^4)$, $\kappa_5 = (5/2)(a_1^2 + 3a_2^2/4)a_1^2 a_2 \sin(\phi_2)$. These expressions show that, in the event that $a_2 = 0$, an appropriate estimate of κ_4, when tranformed, will provide an estimate of a_1, the primary parameter of interest. An estimate of the noise variance can then be obtained from an estimate of κ_2, though it need not exist for each sample.

More generally, we use unbiased estimates $k_1, k_2, \ldots, k_{12}$ of the corresponding cumulants to estimate all of the parameters and, by propagation–of–error, their approximate standard errors. Estimates are obtained following an approximate test for the existence of the harmonic term (the procedure accounts for the possibility that, if the harmonic is in phase ($\phi_2 = 0$), testing for nonzero values of $\kappa_3, \kappa_5, \ldots$ is not sufficient to detect the harmonic). In the worst case ($a_2 > 0, \phi_2 = 0$), the sixth cumulant is also needed in the derivations. Estimates of higher–order cumulants are required because the standard deviation of k_j is a function of κ_{2j} and lower order cumulants.

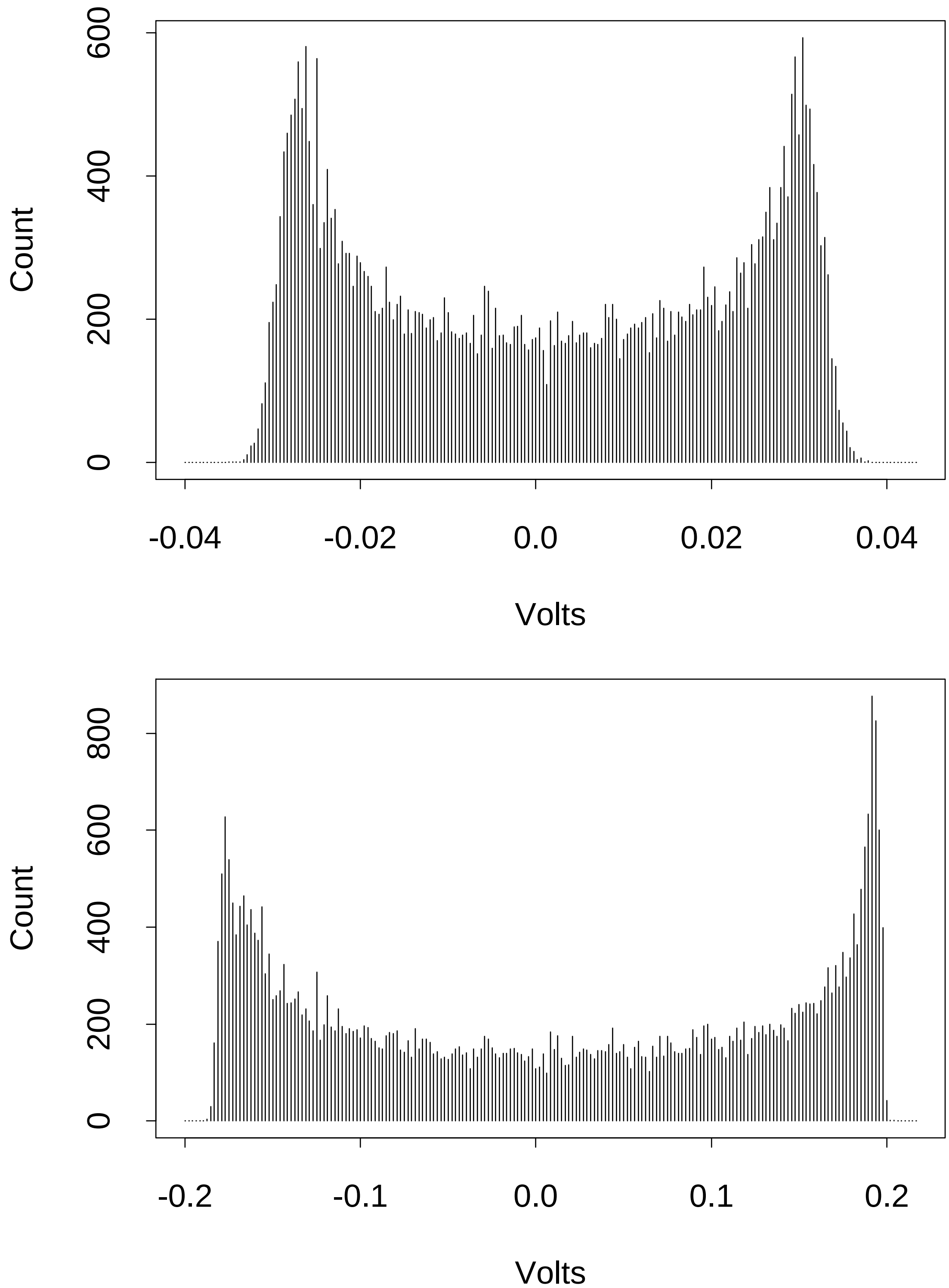

Figure 7: Histograms of 40,000 random time–samples from a 3 GHz waveform. The histogram in the top figure, by its symmetry, would suggest that a second harmonic, if present, is in phase with the fundamental. Asymmetry of the bottom histogram, which was obtained from a signal at a higher power level, is consistent with a second harmonic term. Measurable harmonic content is more likely as the power level is increased.

3.1.8. Error Analysis of Interferometric Retardance Measurements

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A NIST effort to develop an accurate and stable retardance SRM has necessitated the development of measurement methods for optical retardance. Retardance is a property of devices commonly known as waveplates, which are used for polarization control. Three methods have been developed. Two methods rely on polarimetric techniques. The third one is based on an interferometric technique that exhibits different error sources and complements the polarimetric measurements.

The retarder is a double–rhomb design. The largest measurement uncertainty arises from the reflectance of the rhomb faces. This is because the laser used in this measurement system has a long coherence length, multiple reflections from the rhomb faces can interfere coherently and cause variations in retardance measurements. The error in retardance due to coherent reflections is given by

$$Y = Y(r,\, \delta_0,\, U) = \tan^{-1}\left(\frac{-r\sin(U+\delta_0)}{1-r\cos(U+\delta_0)}\right) - \tan^{-1}\left(\frac{-r\sin(U-\delta_0)}{1-r\cos(U-\delta_0)}\right)$$

where r is the reflectivity, δ_0 is the retardance of rhomb, and U is a random variable and is uniformly distributed over the interval $(0,\, 2\pi)$. The pdf of Y is found to be

$$f_Y(y) = \frac{(1-r^2)\sin(\delta_0)}{\pi|\sin(\delta_0-y)|\sqrt{4r^2\sin^2(\delta_0-y)-[\sin y - r^2\sin(2\delta_0-y)]^2}},$$

$$\tan^{-1}\left(\frac{r^2\sin(2\delta_0)-2r\sin\delta_0}{1+r^2\cos(2\delta_0)-2r\cos\delta_0}\right) < y < \tan^{-1}\left(\frac{r^2\sin(2\delta_0)+2r\sin\delta_0}{1+r^2\cos(2\delta_0)+2r\cos\delta_0}\right).$$

It can be shown that, for a wild range of δ_0, the mean of Y is 0 and the standard deviation of Y is proportional to the reflectivity r.

The double–rhomb retarder has endfaces with reflectance r_a and an internal interface with reflectance r_b. The total retardance error, resulting from multiple reflections between the endfaces and between the internal interface and endfaces, is given by

$$Z = Y(r_b,\, \delta_0/2,\, U_1) + Y(r_b,\, \delta_0/2,\, U_2) + Y(r_a,\, \delta_0,\, U_1+U_2)$$

where U_1 and U_2 are independent uniform random variables over the interval $(0,\, 2\pi)$. If δ_0 is close to $90°$, it can be shown that the mean of Z is 0 and the variance of Z is well approximated by $2(r_a^2 + r_b^2)$.

The results indicate that the noise is zero–mean and anti–reflection coatings should be applied to rhomb faces to reduce the variation. A manuscript, describing the interferometric system and the detailed error analysis, has been submitted to *Applied Optics*.

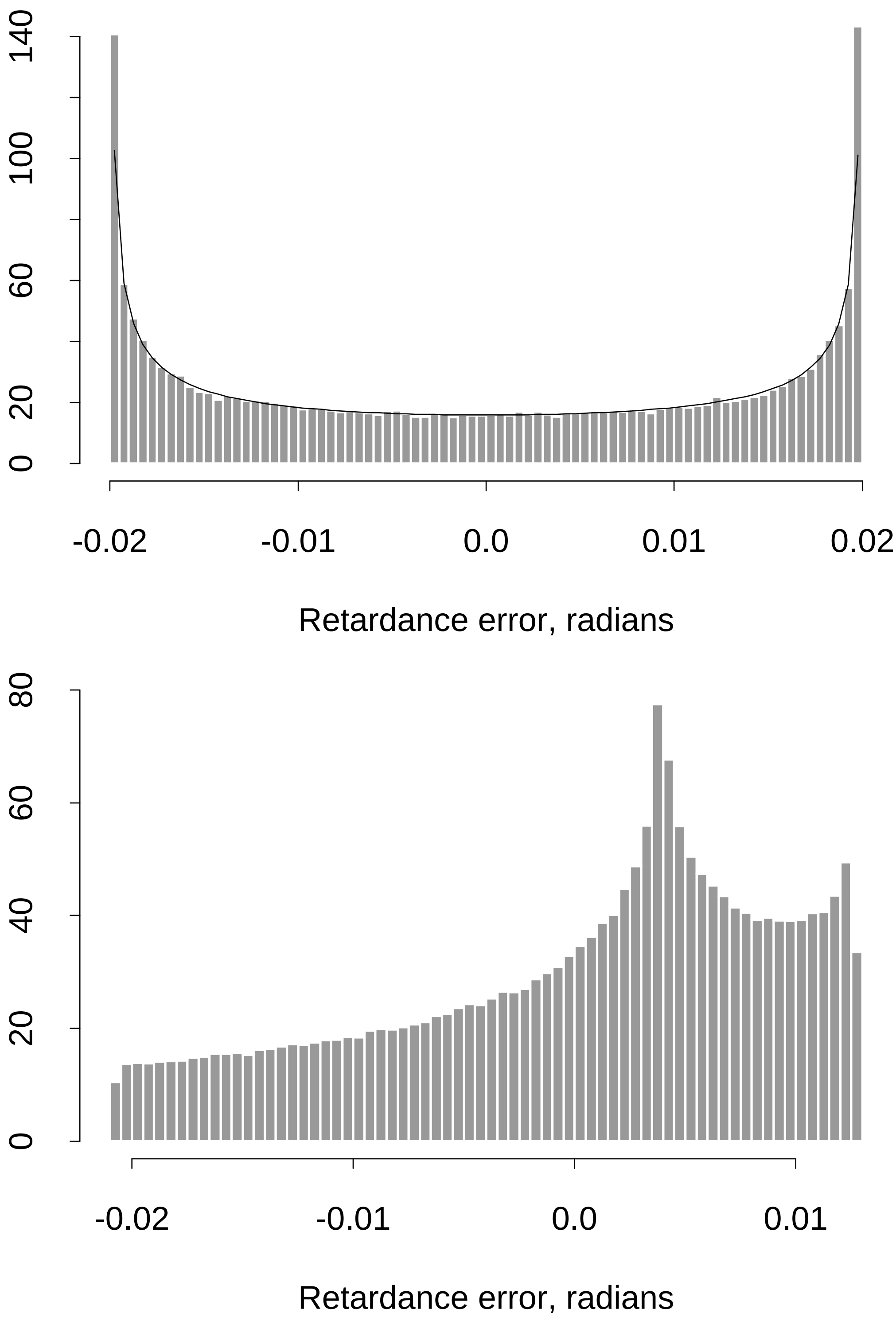

Figure 8: The top figure displays the sample (based on 100000 simulated values of U) and population (solid line) pdfs of Y with $r = 0.01$ and $\delta_0 = 91°$. The bottom plots the sample pdf of Z (based on 500000 simulated values of U_1 and U_2) with $r_a = 0.002$, $r_b = 0.006$ and $\delta_0 = 89°$.

3.1.9. Wavelength Calibration

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Many new high capacity systems use several laser transmitters, operating at slightly different wavelengths, to increase the transmission capacity of a single fiber, a technique known as wavelength division multiplexing (WDM). This requires that the wavelengths of the individual lasers be well known and controlled. A NIST–developed SRM is a fiber–connected gas absorption cell that permits quick wavelength calibration of instruments, such as optical spectrum analyzers, used in the development of WDM systems. The measurements used in the calibration consist of wavelength and corresponding absorption power. The calibration is carried out by fitting a model, called Voigt, to the data.

A Voigt density is obtained by convolving a Gaussian density with a Lorentzian (Cauchy) density. Specifically, if U is a Gaussian random variable with parameters μ and σ, and V is a Lorentzian random variable with parameters μ and λ and is independent of U, then $W = (U + V)/2$ is distributed as a Voigt with density function given by

$$f(w) = \frac{2\lambda}{\pi^{1.5}} \int_{-\infty}^{\infty} \frac{\exp(-u^2)}{\lambda^2 + \left[2(w-\mu) - \sqrt{2}\sigma u\right]^2} du.$$

For data–fitting purpose, a Voigt model must be general enough to allow for an arbitrary translation of the data. The Voigt model, relating absorption power (y) and wavelength (x), is given by

$$y = \beta_0 + \beta_1 \int_{-\infty}^{\infty} \frac{\exp(-u^2)}{\beta_2^2 + [2(x-\beta_3) - \beta_4^2 u]^2} \, du$$

where β_0, $\beta_1, \cdots, \beta_4$ are parameters of the model. The parameters of interest are the wavelength that attains the maximum absorption power (β_3), and the relative height and width of the absorption power spectrum (functions of β_2 and β_4). Since both power and wavelength are subject to measurement errors, a Fortran program, utilizing a nonlinear errors–in–variables regression procedure, has been developed to estimate the parameters and their standard errors.

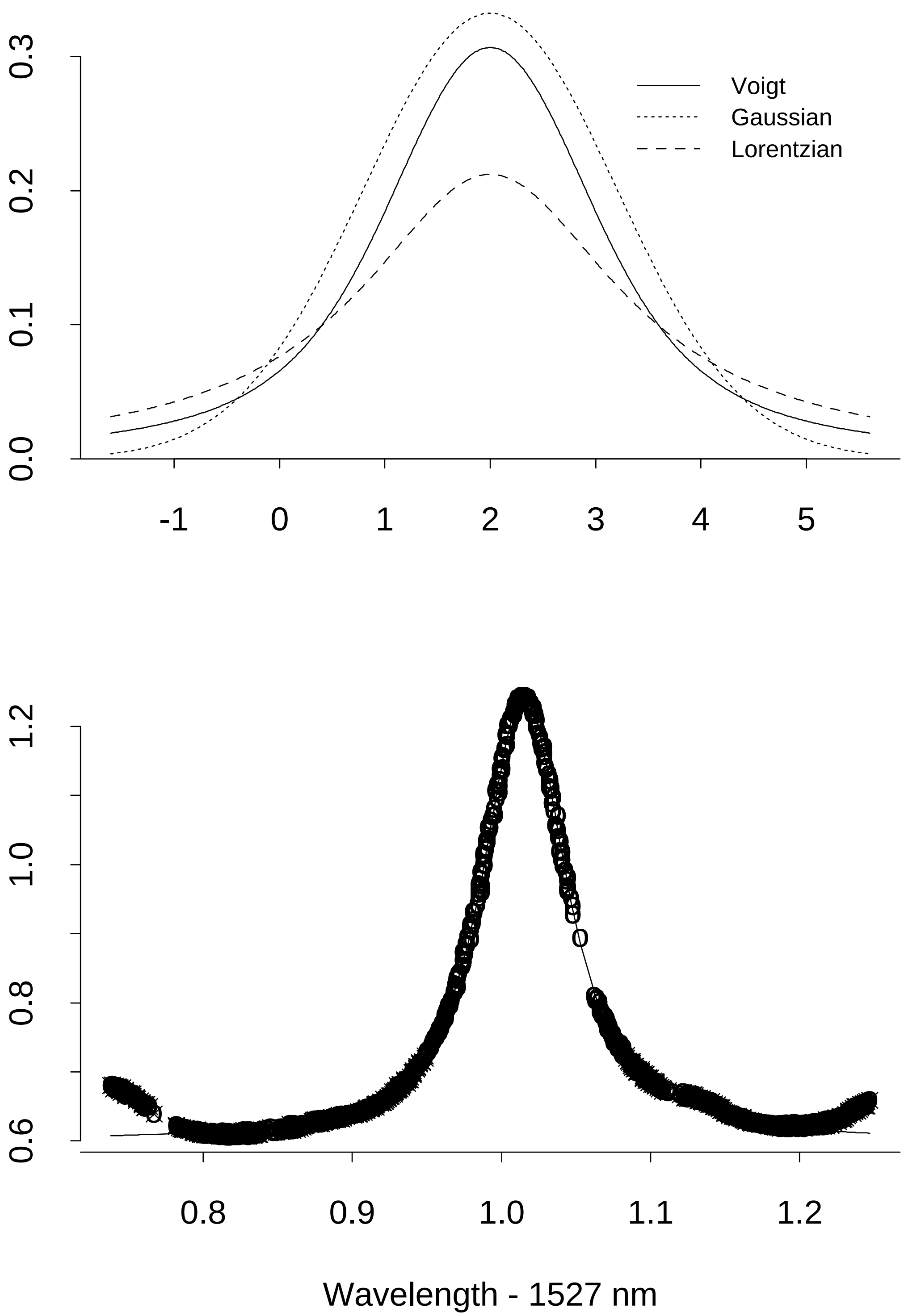

Figure 9: The top figure displays the density functions of Gaussian, Lorentzian and Voigt ($\mu = 2$, $\lambda = 1.5$, and $\sigma = 1.2$). It shows that a Voigt possesses the peak feature of a Gaussian and the tail profile of a Lorentzian. The bottom plots the scatterplot of wavelength vs. power and the Voigt (solid curve) model fitted by the errors–in–variables regression. The "o" points were used to fit the model and "×" points were not used.

3.1.10. Estimating The Measurement of Pitch in Metrology Instruments

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NIST is in a process of developing a new low-accelerating-voltage scanning electron microscope (SEM) magnification calibration reference standard 2090. This standard will be useful for all applications in which the SEM is currently being used, but it has been specially tailored for many of the particular needs of the semiconductor industry. In order for the NIST certification process to be complete, an estimate of the pitch measurement and its uncertainty must be evaluated. As the precision and accuracy of metrology instruments are pushed to the nanometer level, the evaluation of the performance of the pitch measurement algorithm becomes increasingly important. Figure 1 shows the diagram of the NIST SRM 2090a prototype SEM magnification standard. The left hand-side is a lowest magnification drawing showing the 3 mm and 1 mm pitch patterns, while the right hand-side of Figure 1 is at high magnification showing the two 4 micrometer (μm) and eight 0.2 μm pitch structures as well as the focusing and astigmatism-correction crosses.

The prototype SRM 2090a data was obtained by using the NIST SEM-based metrology system. A pitch distance between two pitch structures is defined as the distance between the left (or right) edge of one pitch structure and the left (or right) edge of another pitch structure. Mathematically, when the SEM signals at the edges are parallel straight lines the pitch distance is uniquely defined. However, in reality, when measurements are done by an SEM system as described above, the edges formed by discrete data points are not necessarily parallel.

Traditionally, a least squares regression line is fitted to the data points corresponding to each of the left (or right) edges of a pitch structure. Then, the distance between the two fitted lines (corresponding to two left or two right edges) at a certain height on the vertical axis is assigned as the pitch distance between the pitch structures. A disadvantage for this approach is that the pitch distance varies with the height at the vertical axis because in general these two fitted regression lines are not parallel. Another disadvantage for the traditional algorithm is that it is difficult to estimate the uncertainty of the pitch distance. We developed a statistical model based algorithm to eliminate this kind of uncertainty. The estimator of pitch distance and its uncertainty have been derived. Evaluations based on simulations show that the uncertainty of measurement of the pitch distance by the new method is smaller than that by the traditional one.

This work has been presented by Nien Fan Zhang at the SPIE's (The International Society for Optical Engineering) 1996 International Symposium on Microlithography.

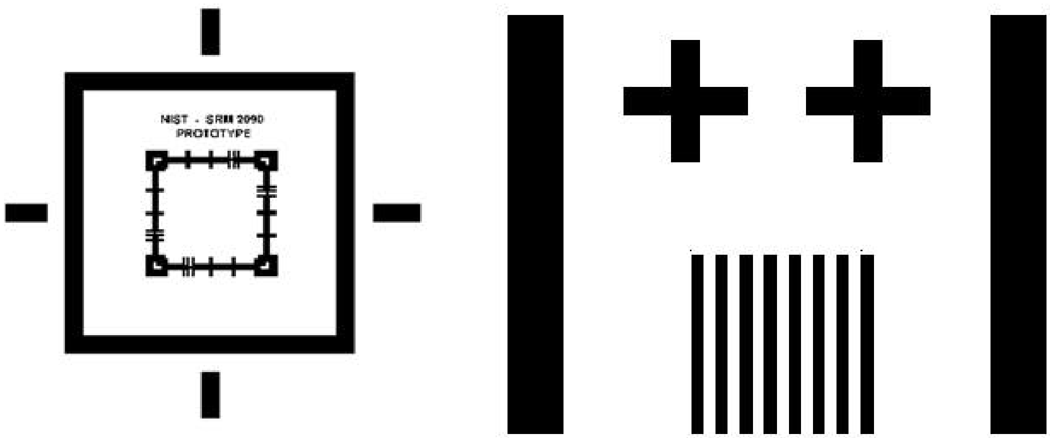

Figure 10: This figure shows the diagram of the SRM 2090a prototype SEM magnification standard.

3.2. Experiment Planning and Interpretation

3.2.1. Stochastic Modeling of Aerosol Trajectories

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Under the auspices of the 1980 Japan-US Science pact, Kensei Ehara of Japan's National Laboratory of Metrology visited NIST. During this visit, he proposed a new kind of spectrometer which separates aerosol particles according to their mass to charge ratio. Aerosol particles are injected into the space between two corotating cylinders. A voltage difference is applied between the cylinders. Electrical and centrifugal forces act on the particles. Further, Brownian motion effects are significant for particles with small mass. In earlier work (in collaboration with C. Hagwood and A. Negiz), we computed the probability that a particle of a given mass and diameter will pass through the spectrometer or stick to either the inner or outer cylinder wall. This probability, i.e. the transfer function, depends on the adjustable rotational rate and applied voltage.

In new work, we estimate the concentration of aerosols within the spectrometer. The space between the inner and outer walls of the spectrometer is discretized into pixels. For a given random initial position at the inlet boundary, a trajectory is computed. When a trajectory enters a particular pixel, the cumulative visitation time for that pixel is updated. Based on many random trajectories, a visitation time histogram is computed. By normalizing this histogram, we estimate the spatial concentration distribution.

Near the boundaries, the concentration is complex. Typically, when solving the diffusion equation for concentration, simple boundary conditions on concentration are typically assumed. Hence, the new work suggests that such simple boundary conditions are not appropriate.

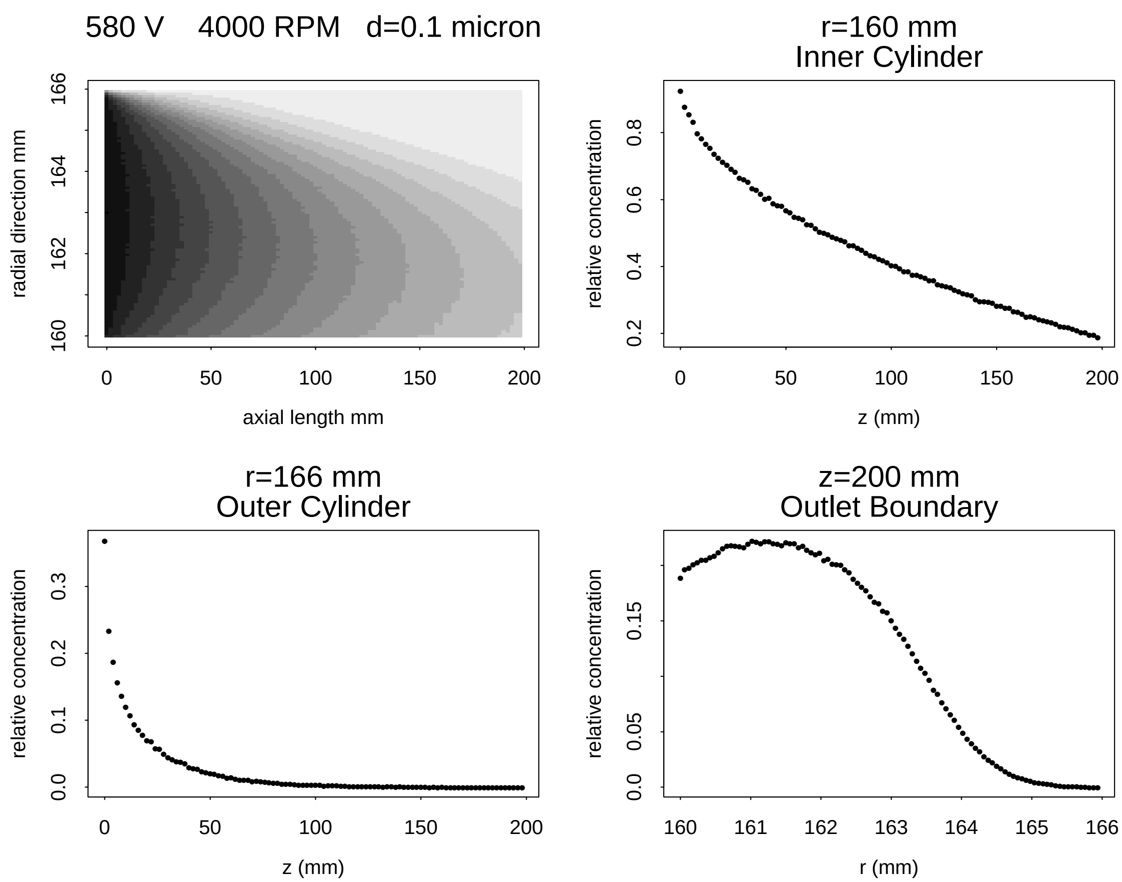

Figure 11: Upper left: computed relative concentration of spherical aerosol particles with diameter 0.1 micron and density of 1g cm^{-1}. The radii of the cylinders are 160 and 166 mm. The axial length of the cylinders is 200 mm. The rotation rate is 4000 RPM and the applied voltage is 580 V. The other plots show estimated concentration near the boundaries. At the inlet boundary, the initial concentration is uniform.

3.2.2. Magnetic Trapping of Ultra Cold Neutrons and Determination of the Mean Lifetime of the Neutron

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Researchers from Harvard University, Los Alamos National Laboratory, University of Washington, University of Berlin, and NIST plan to produce and confine polarized Ultra Cold Neutrons (UCN) in a magnetic trap. Based on this new technology, the neutron lifetime will be determined at a precision up to 100 times better than the current value. Along with other experimental data, a measurement of the mean lifetime of the neutron allows one to test the consistency of the standard model of electroweak interactions. Further, the mean lifetime of the neutron is an important parameter in astrophysical theories. Statistical and computational work has focused on optimal experimental design and dynamical studies of marginally trapped neutrons.

Optimal Estimation. There will be many run cycles of a two stage experiment. In the first stage of each run, neutrons from the NIST Cold Neutron Research Facility are guided into a superfluid ^{4}He bath where they dissipate almost all their energy by inelastic scattering. These UCN are confined in a magnetic trap. After filling the trap to some level, the neutron beam is blocked and decay events, as well as background events, are recorded. Denote the duration of each stage as T_{fill} and T_{decay}. Two algorithms for estimating the mean lifetime are compared. In one method, the event time data is summarized as a histogram. The time endpoints of the histogram are selected so that the expected number of counts per bin contributed by the decay process, is constant. In the second method, the lifetime is estimated from the complete sequence of event times. The histogram method yields a less variable estimate of the mean lifetime. The optimal strategy for time allocation is found by minimizing the asymptotic variance of the lifetime (estimated from the pooled histogram data from all cycles) as a function T_{fill} and T_{decay}, given knowledge of the filling rate of the trap and parameters which characterize the background process. The validity of the asymptotic approximation is demonstrated in Monte Carlo experiments.

Marginally Trapped Neutrons. Neutrons with sufficiently high energy escape the trap, but not immediately. These "marginally trapped" neutrons may decay before escaping. In a Monte Carlo study, many trajectories are simulated. For each trajectory, we compute the escape time. Beyond about six seconds of elapsed time, we find that escape times can not be predicted in a numerically stable manner.

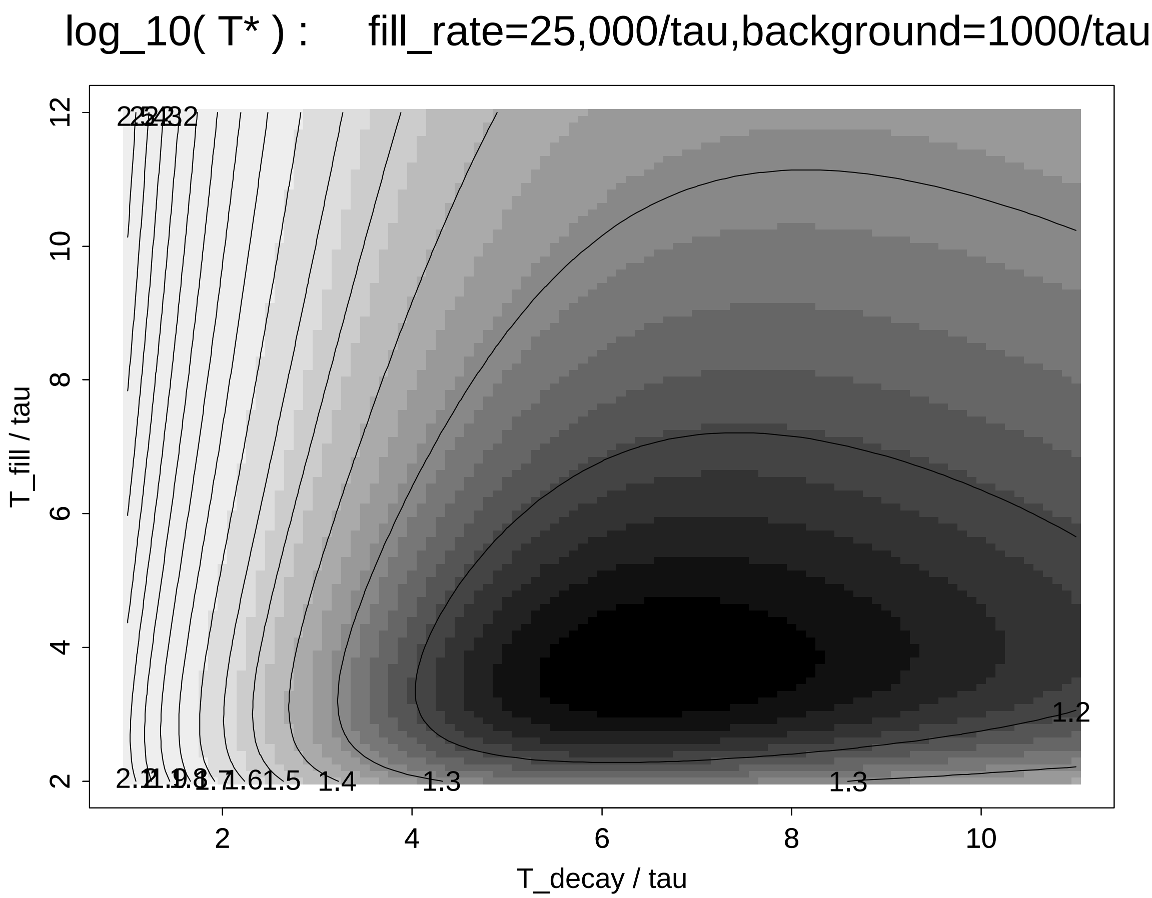

Figure 12: The approximate mean lifetime of the neutron is $\tau \approx 890\ s$. During the fill stage of each run cycle, the expected number of confined neutrons grows as

$$\lambda_{fill} * \tau(1 - exp(-T_{fill}/\tau))$$

where λ_{fill} is the rate at which neutrons enter the trap, τ is the mean lifetime of the neutron and T_{fill} is the duration of the fill stage. We express the asymptotic standard error of the mean lifetime, estimated from data pooled from all run cycles, as

$$\frac{\sigma_{\hat{\tau}pool}}{\tau} \approx 0.001\sqrt{\frac{T^*}{T_{total}}}$$

where the duration of the entire experiment is T_{total}. Above, $log_{10}(T^*)$ is plotted as a function of T_{fill}, T_{decay} for the case where $\lambda_{fill} = 25,000/\tau$ and the background is a stationary Poisson process with intensity rate equal to $1000/\tau$.

3.2.3. Detection and Quantification of Isotopic Ratio Inhomogeneity

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Most chemical elements in nature are multi-isotopic; i.e. they exist in several atomic forms with the same number of protons but different number of neutrons in their nuclei. Geologic and biological processes can alter the isotopic ratio of particular isotopes in a sample. Also, isotopic ratios can be intentionally altered by enrichment schemes. Materials with constant isotopic ratios are said to be isotopically homogeneous. In an inhomogeneous material, the isotopic ratio varies from location to location.

We quantify the spatial variation of the ratio of two isotopes within a material based on Secondary Ion Mass Spectrometry (SIMS) data. At many spatial locations, a detector counts each of two isotopes of a chemical element. At each location, we predict the less abundant isotope count in terms of the measured value of the more abundant isotope count and the estimated mean isotopic ratio. The difference between the measured and predicted value is divided by an estimate of its standard deviation. The approximate standard deviation of the prediction error is computed by the propagation of the errors method. To estimate the spatial standard deviation of the isotopic ratio, we equate the sum of squared standardized residuals to its approximate expected value. The approximate expected value is obtained by a bootstrap resampling method. Based on the estimated null distribution of the estimated standard deviation, we test the hypothesis that the isotopic ratio is constant throughout the sample. To check the validity of our methods, we analyze SIMS data collected from a homogeneous chromium sample. Results are consistent with the hypothesis of homogeneity. We simulate data corresponding to a sample where the isotopic ratio has a binary distribution. We find that when the standard deviation of the binary distribution exceeds twice the 86th percentile of the null distribution, detection of inhomogeneity is almost certain. Further, the estimated standard deviation closely tracks the actual standard deviation.

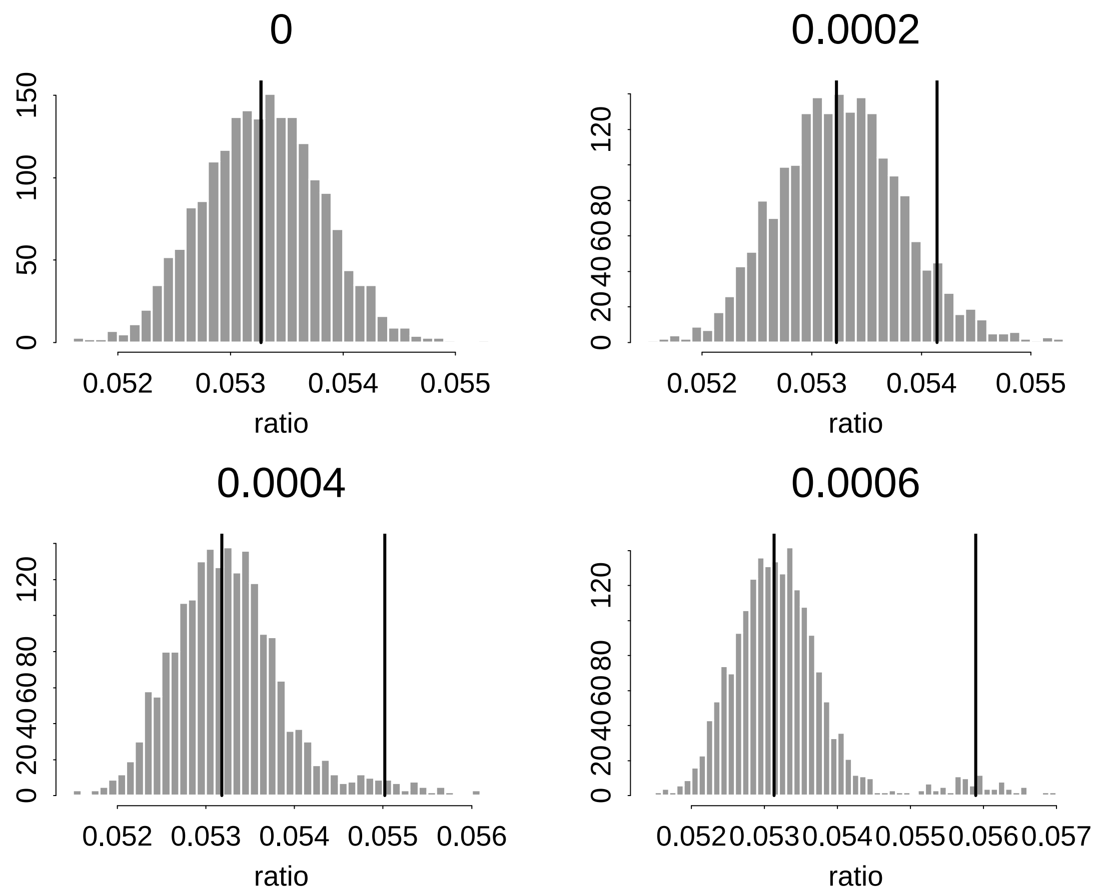

Figure 13: Sample histograms corresponding to simulated data where the isotopic ratio has a binary distribution. The standard deviation of the mixture distribution σ_r varies from 0 to 0.0006. The solid lines correspond to the values of the two isotopic ratios in the mixture. Mixing fractions are 0.95 and 0.05. For $\sigma_r > 0.0002$, for a test with size 0.10, the detection rate (of inhomogeneity) exceeds 99 percent and the estimated standard deviation $\hat{\sigma}_r$ closely tracks σ_r.

3.2.4. Modeling Constitutive Behavior of Steels

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Scientists in the Materials Reliability Division seek to improve the quality of sheet metal products manufactured by hot-strip rolling. The project is funded by the American Iron and Steel Institute and the Department of Energy. To achieve this goal, it is necessary to understand how a metal deforms under high stress. Based on experimental data collected at NIST, SED is developing a statistical model for predicting stress-strain behavior of metals as a function of chemistry, grain size, temperature and initial strain rate. The current model is an improved version of an earlier model developed at NIST.

The prediction variables in the new model are normalized so that the relative contribution of the different sources of variability are apparent. In the model, there are two terms in the prediction for stress. The first prediction term is a monotonically increasing function of strain. The second term represents a correction due to the dynamic recrystallization of the material. Due to this effect, stress is not necessarily a monotonically increasing function of strain.

Due to the high number of parameters in the model, the estimated parameters were obtained using a regularization approach. The model parameters are determined by minimizing a loss function which is the weighted sum of two terms. The first term is the sum of squared residuals. The second term is a penalty function. The model predicts the asymptotic value of stress for large values of strain. The penalty function is large when the predicted asymptotic value of stress is far from a prior estimate of the aysmptotic value. A weighting factor determines how much influence the penalty function has, relative to the sum of squared residuals term, in determining the parameter values. Estimates were obtained for various values of the weighting factor. Scientific judgement was used to select the best value of the weighting factor.

The new model has better theoretical properties than does the earlier model. For certain choices of initial strain rate and temperature, the stress-strain curves should satisfy monotonicity constraints. For low to moderate strains, as strain is increased, the predicted stress curves for different grain sizes should not cross. However, the predicted stress curves from the older model did cross. In contrast, the predicted stress curves from the improved model do not cross.

In coming months, data collection and validation studies will be based on formal statistical planning to further refine or replace stress-strain models.

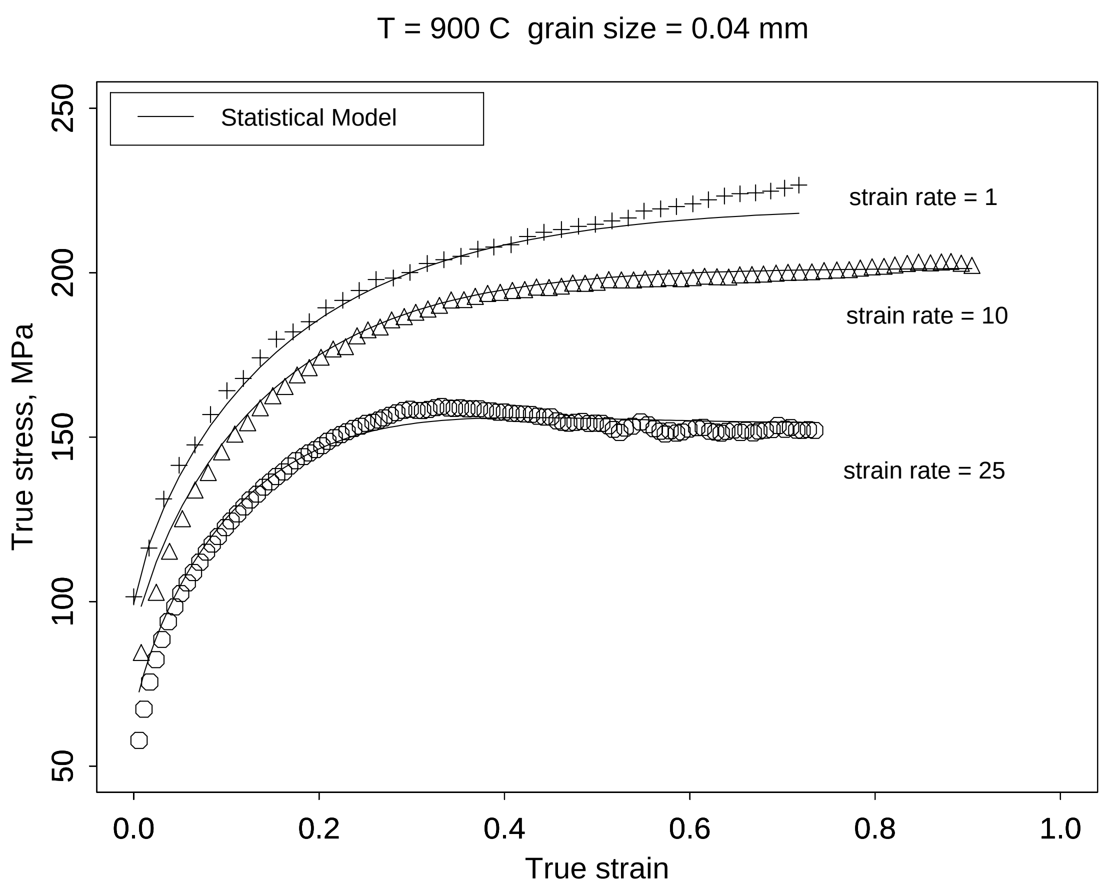

Figure 14: The measured stress-strain curves and a statistical model for the curves are compared for different experimental conditions. Due to dynamic recrystallization, strain is not a monotonic function of stress.

3.2.5. Consistency of Secondary Ion Mass Spectrometry and Neutron Depth Profiling Measurements

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Neutron Depth Profiling (NDP) is a nondestructive method for analysis of the concentration profile of an element in material. Inferences about the concentration depth profile are based on the observed energy spectrum of charged particles emitted due to specific nuclear reactions. The detector response function (DRF) is a probability transition matrix which relates the depth of emission to the expected energy spectrum of the detected particles. The DRF depends on the geometries of the emitter and detector, and assumed models for the stopping power of the material, energy straggling, multiple scattering and random detector measurement error and detector calibration. In previous work, we developed a computer code to predict the DRF.

We check the consistency and validity of the NDP method as follows. The depth profile of boron in a silicon sample was measured by Secondary Ion Mass Spectrometry (SIMS). In a separate experiment, the NDP energy spectrum was measured for the same sample. Based on the measured SIMS profile and the modeled DRF, we predict the NDP energy spectrum.

We attribute the observed differences in the predicted and observed NDP spectra to imperfect knowledge in one or more of the following: stopping power of silicon, density of silicon, calibration of NDP detector, energy resolution of the detector, straggling in silicon, and calibration the SIMS instrument. Based on the current data, we can not resolve which of the factors is responsible for the discrepancy. To better explain the discrepancy, a new experimental study is underway.

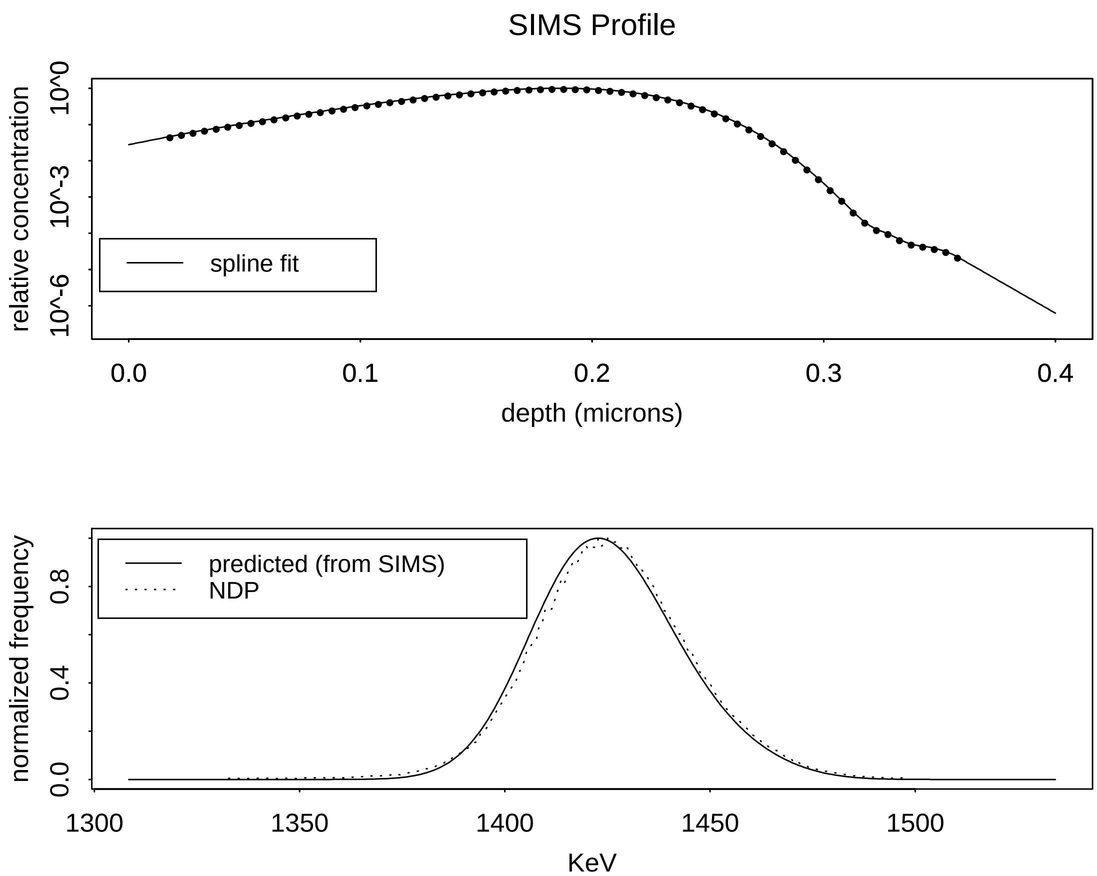

Figure 15: Top: The concentration profile of boron in silicon measured by SIMS. This profile is fit with a smoothing spline. We convolve the fitted concentration profile with the modeled detector response function to get a predicted energy spectrum. Bottom: Predicted and observed NDP energy spectra.

3.2.6. NIST Ceramic Machining Consortium

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Said Jahanmir
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In July of 1992, a research program on ceramic machining was initiated by the Materials Science and Engineering Laboratory prompted by the results of a comprehensive survey of U.S. industry which confirmed that the primary impediment to the widespread use of advanced ceramics is the high cost of machining. To assure that industry needs were properly addressed and to take advantage of expertise existing at other research institutions, NIST established a consortium with members from industry, academia, and government. Currently, their are 23 active members. The mission of the program is to assist U.S. industry in the development of precision machining for the manufacturing of cost-effective advanced ceramic products.

A primary goal of the consortium is to collect and analyze data on the effect of grinding parameters on ceramic properties/performance. Of particular interest is how the strength and surface integrity vary with the grinding parameters, and the determination of grinding conditions that result in high material-removal rates without significantly decreasing the strength of the ceramic.

An experiment involving eight different commercial and industrial grinding facilities was designed to evaluate the influence of grinding conditions on the flexure strength of three ceramic materials: sintered reaction bonded silicon nitride (SRBSN), reaction bonded silicon nitride (RBSN) and sintered silicon nitride (SSN). Given the overall goals of the experiment and the constraints due to time, money and the available material, a full factorial in four primary factors partially confounded incomplete block design was constructed and implemented. The grinding parameters varied were table speed, down feed, grit size and the direction of grinding with respect to the tensile direction of the four-point bend flexure specimens used in the study. A relatively high removal rate grind regime was investigated.

The results showed that, even under the most aggressive grinding conditions, no significant change in flexure strength was measured when grinding was parallel (longitudinal direction) to the tensile direction. All conditions resulted in a decrease in flexure strength when grinding was perpendicular (transverse direction) to the tensile direction. In addition, in the transverse direction, there was a significant effect due to wheel grit size and an interaction between table speed and down feed (for SRBSN). The other two materials had similar conclusions.

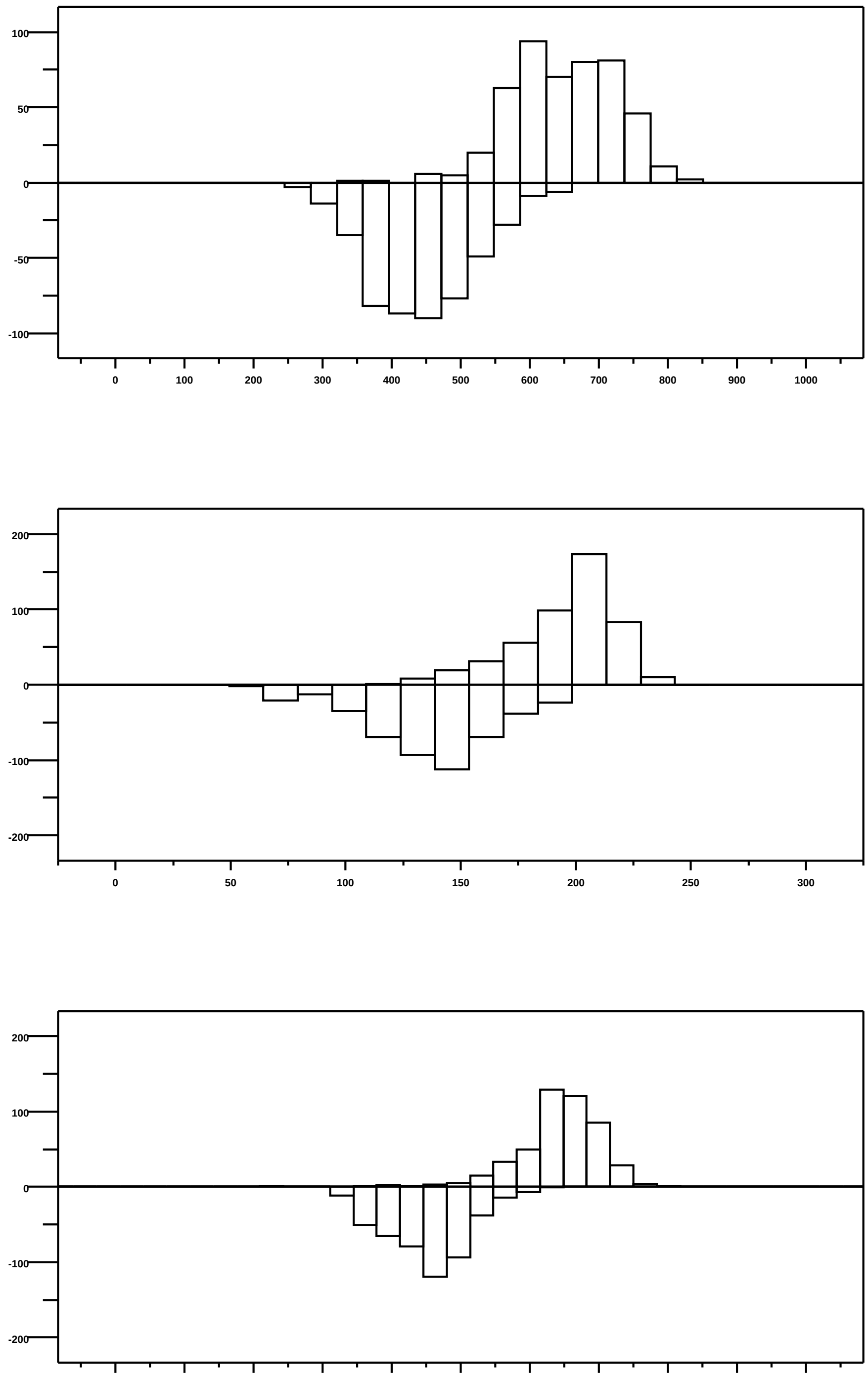

Figure 16: Bihistogram of Longitudinal Grinding Direction versus Transverse Grinding Direction for SRBSN, RBSN, SSN (top to bottom). The shift in the histogram represents the effect direction has on the strength of the ceramic. The effect is evident in all three materials and in the same direction; that is, transverse grinding resulted a lower flexure strength.

3.2.7. Fabric Ignition Propensity of Cigarettes

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Richard Gann
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Cigarette ignition of soft furnishings (upholstered furniture and bedding) has long been the leading cause of fire deaths in the United States. In 1984 and 1990, Federal legislation directed research efforts to determine whether the potency of cigarettes as an ignition source could be moderated. Under this legislation NIST produced a test procedure known as the Mock-up Ignition Test Method, which was designed to distinguish the propensity of different types of cigarettes to ignite soft furnishings.

Subsequently, the Mock-up Test has been criticized for the use of a test fabric, cotton duck, that is not indicative of the performance of fabrics used in the manufacture of upholstered furniture. A joint venture of cigarette industry firms purchased approximately 500 upholstery fabrics and used them to test 4 experimental cigarettes. They concluded that most fabrics ranked cigarettes differently from the cotton duck used in the Mock-up Test.

SED statisticians were called upon to re-analyze the data from the industry-sponsored study. Using several parallel modeling procedures, we demonstrated that there was an interaction between the fabric and the ignition propensity of the cigarettes. However, we distinguished two types of interactions. In the first type, the relative magnitudes of the ignition propensity vary among the fabrics, but the rankings of the cigarettes do not. In the second type, the rankings also vary. The first type of interaction would not invalidate the Mock-up Test, whereas the second one would.

In order to determine which of the two interactions existed, we defined a consistency score that measured the agreement with the cigarette rankings from the Mock-up Test. Positive scores indicate agreement and negative scores indicate disagreement. Most of the results from the 500 fabrics were not applicable to the analysis because the fabrics either always ignited or never ignited during pre-testing or testing. Owing to the power of the study, only 41 of the 79 the applicable fabrics statistically distinguished the cigarettes.

The figure displays a histogram of the positive scores (given in the top portion of the plot) and negative scores (given inverted in the bottom portion of the plot). The 41 fabrics that significantly discriminate among the 4 cigarettes at the 5% level are emphasized with a filled square. Note three significant patterns in the figure: (1) there are substantially more fabrics with a positive score than a negative score; (2) the positive scores tend to be larger in magnitude than the negative ones; (3) considering only the fabrics that significantly discriminate, the first two notes are still true. We concluded from this analysis that, although there is an interaction between fabrics and cigarettes, for most fabrics the rankings of the cigarettes are consistent with the Mock-up Test.

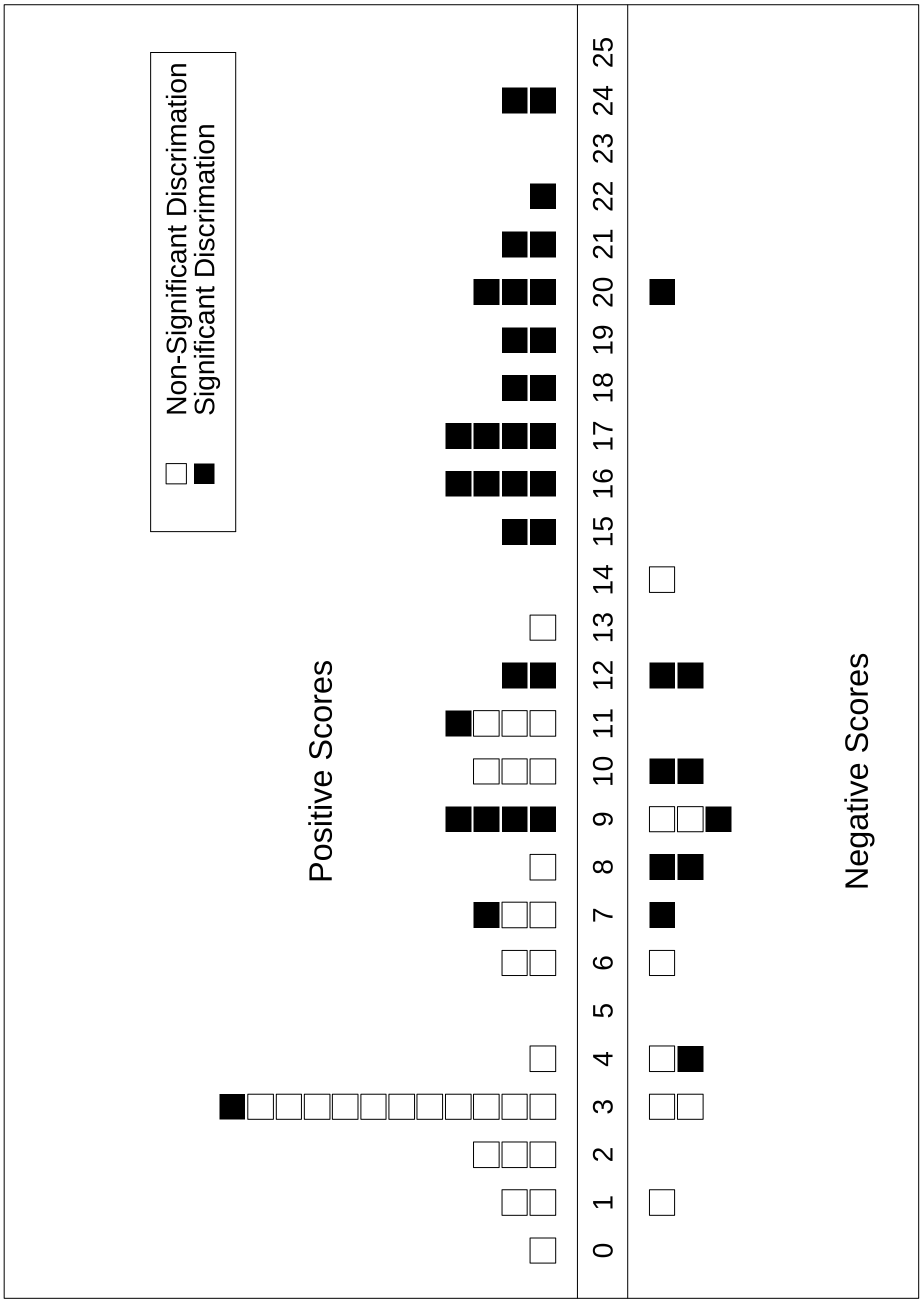

Figure 17: Consistency scores.

3.2.8. Creep-Rupture Performance of Adhesively-Bonded Roofing Seams

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Adhesively-bonded EPDM (a rubber material) is widely used for low-slope industrial roofing. There are two main types of adhesive systems for seams on these roofs: a liquid adhesive, and several varieties of tape adhesive. Liquid adhesive is widely used, but it is volatile and relatively expensive to apply. An objective demonstration that tape adhesives are at least as reliable as the liquid will greatly increase the use of these adhesives. A consortium of NIST, professional roofing trade associations, and roofing adhesive manufacturers was formed, in part, to perform such a study.

The chosen measure of performance for the experimental seams is creep lifetime; i.e. the time-to-failure under a constant load. In Phase I of this investigation, specimens from two tape systems and a liquid adhesive were tested in creep-rupture at various loads. The main conclusion of this phase was that the tape-bonded seams appear to perform at least as well (in terms of creep life) as adhesive-bonded seams.

Phase II of this investigation concerns the performance of tape-bonded seams under laboratory simulations of field preparation conditions. A 2^{5-1} fractional factorial experiment involving application factors was designed and analyzed, for each of 2^2 combinations of material factors. The application factors are

Factor	Level 1	Level 2
Primer	Not Primed	Primed
Surface Condition	Clean	Contaminated
Application Pressure	Low	High
Application Temperature	Low	High
Time-at-Temperature	Short	Long

The material factors are two tape systems, each obtained at a thickness that is typical of what is used in practice, as well as a lesser thickness.

The figure displays the means of eight replicates for ordered factor combinations, with the material factors indicated by plot symbols. The average lifetime for liquid adhesive specimens is taken from Phase I of this study. It appears that if tape systems are well prepared (primed, cleaned, etc), then tape-bonded seams can be expected to have creep-life at least comparable to that of well-prepared adhesively-bonded seams.

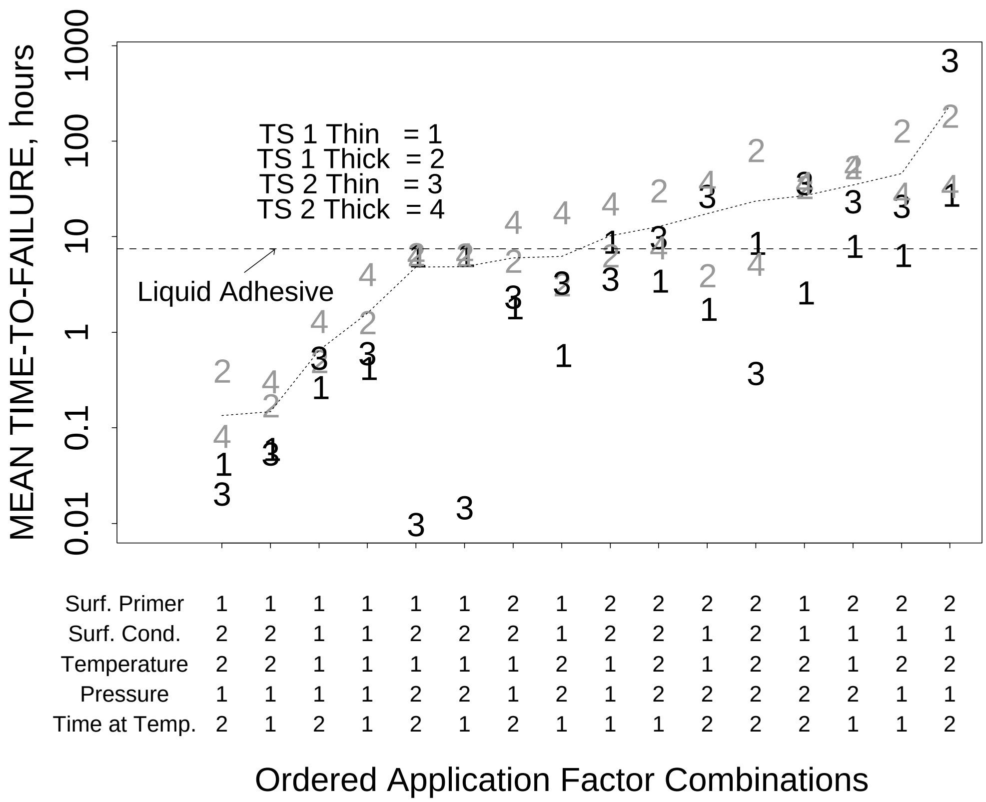

Figure 18: Creep-rupture lifetime of tape-bonded EPDM seams for various combinations of application conditions.

3.2.9. Impact Resistance of Lead-Paint Encapsulants

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Statistical Engineering Division, ITL

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Building Materials Division, BFRL

Lead paint is known to be potentially dangerous to health, particularly for children; consequently it cannot be used for new structures in this country. However, much lead paint still remains in older housing. Since properly removing and disposing of lead paint is an expensive process, it is natural to consider the feasibility of covering lead-painted surfaces with a coating designed to prevent the lead from escaping into the environment. NIST is in the process of evaluating various lead encapsulants which are commercially available. In particular, the experiment reported on here was designed to evaluate the impact resistance of these coatings, and to compare them with ordinary paints.

Steel, plywood, and drywall panels were painted first with a pink undercoat, and then with one of 12 white coatings: coatings 1-6 are unreinforced lead encapsulants, coatings 7-10 are reinforced encapsulants, coating 11 is a latex paint, and coating 12 is an alkyd paint. Two replicate panels were made for each substrate/coating combination, and 100 squares were ruled on each specimen. According to a statistically-designed experiment, randomly-chosen squares on the various panels were impacted with weights of various impact energies (dropped from various heights, under laboratory conditions). Because of the pink undercoat, cracked coatings were usually obvious, and they were regarded as failures. The impact energies were varied so as to attempt to obtain both failures and non-failures for each panel; however this was not always possible. The dataset, not including controls, consists of binary outcomes from 2970 impacts.

A logistic regression model was fit to these data, and this model assumes that the probability of failure is linearly related to impact energy, with an intercept which depends on coating, substrate, and their interaction. One summary of the results of this analysis, shown in the figure, consists of the estimated energy corresponding to 50% probability of penetration, as a function of coating and substrate. The intervals in the figure are approximate 95% confidence intervals. The very wide confidence intervals usually correspond to substrate/coating combinations for which either all impacts were failures, or else for which none were failures. Perhaps the most obvious conclusion to be reached from this summary is that reinforced coatings are more impact resistant than paint, but it is not clear whether the same can be said for unreinforced encapsulants. Also, it appears that steel, perhaps because it is a hard substrate, provides data which are more sensitive to differences among the coatings than the other substrates.

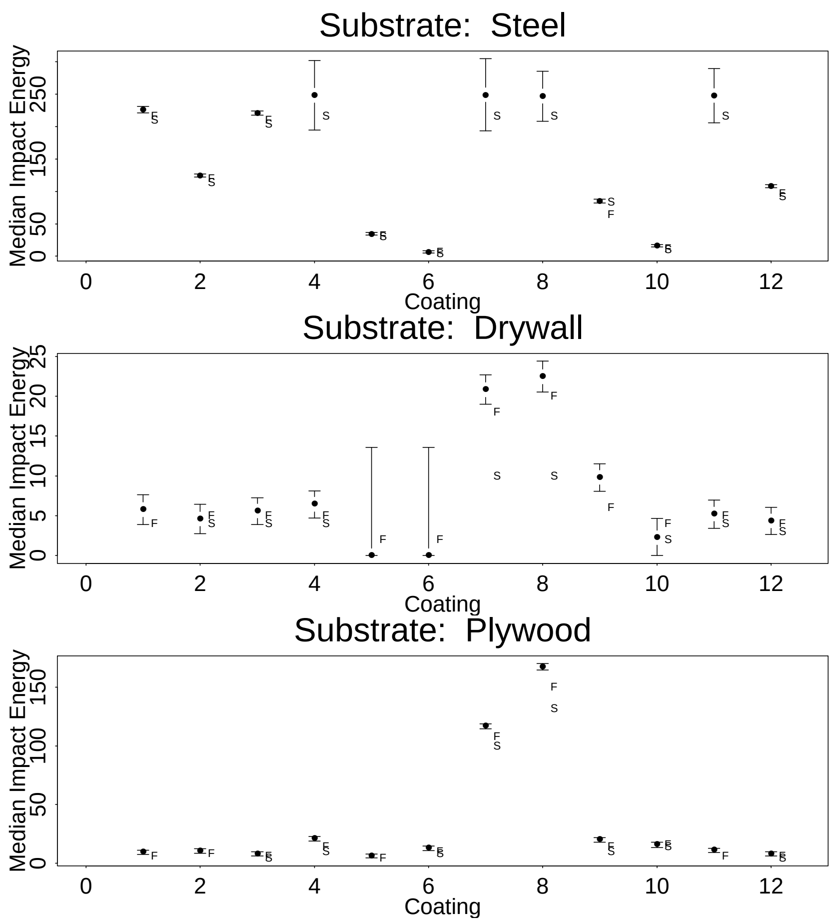

Figure 19: Impact energy corresponding to 50% probability of failure for various coatings and substrates. The plot symbols indicate estimated median failure energies, the smallest (F) energy at which a failure occurred, and the largest (S) energy at which a failure did not occur.

3.2.10. Performance Evaluation for Lead-in-Paint Measuring Devices Under Simulated Field Conditions

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In January 1995, USA Today reported: "Federal Housing officials have ordered retests to detect toxic lead paint in 85 public housing projects where hundreds of millions of dollars in tests may have been flawed. At issue is whether tests by portable X-ray machines are reliable." As part of an effort to improve the reliability of lead-in-paint measuring devices, the U.S. Department of Housing and Urban Development asked NIST to identify and quantify factors affecting the field performance of these portable X-ray fluorescent (XRF) devices. The ultimate objective of this study is to develop a protocol for assessing field precision and bias of XRF instruments that measure lead concentration in painted surfaces.

The protocol would consist of taking XRF measurements on lead-in-paint standards at a specified set of noise conditions known to cause variability in XRF field measurements. The noise conditions are combinations of settings of "noise" factors known to cause variability in field measurements. A candidate list of noise factors can be generated. However, the order of importance of these candidate noise factors, i.e., which cause greatest measurement variability, is currently unknown. A laboratory experiment was conducted to identify the most important noise factors. In this experiment, the noise factors were systematically varied according to a statistical experimental design to study their effect on XRF measurements. Of course, the conclusions from this lab experiment must be validated in the field to ensure that all important sources of variability have been captured. In this experiment, eight noise factors were studied at each of two settings using a 16-run (out of a possible $128 = 2^8$) fractional factorial design. This well-chosen subset of 16 runs permits free and clear estimation of the primary effects of all factors and limited information on two-way interactions between factors. The 16 noise factor conditions were studied for each of four XRF instruments and two lead concentrations.

The figure shows that x6, the distance of the instrument from the surface, is the dominant noise factor, followed by x3, the underlying substrate (wood or steel). Substrate has a large effect for only two of the instruments, indicating that the other two invoke a substrate correction. The "distance-from-surface" noise factor was included to simulate non-flat surfaces such as wood molding, metal pipe, and stucco. A second experiment is being conducted to assess whether "distance-from-surface" is an adequate surrogate for non-flat surfaces, and if so, which distance best reflects the induced variability. The experiment examines non-flat surfaces for several substrates using a full factorial design. The results from these experiments will be used to develop a test protocol for assessing field precision and bias of portable XRF instruments.

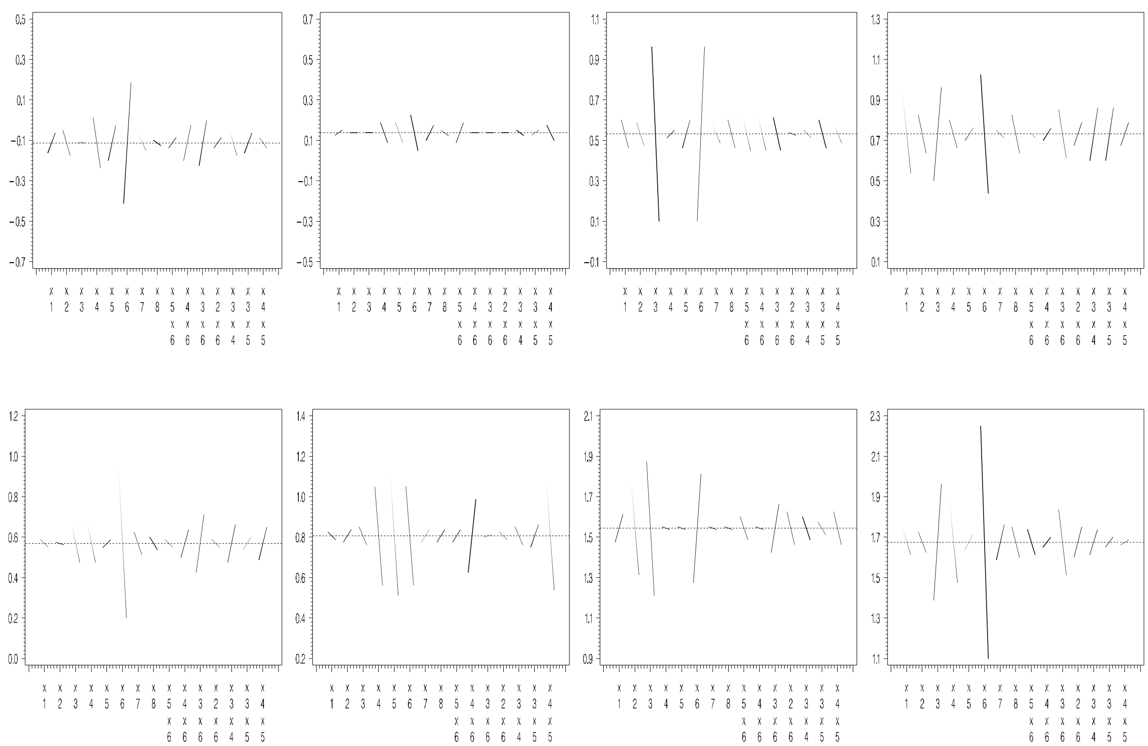

Figure 20: Plots of mean XRF response versus factors for instruments from four manufacturers (columns) and two lead levels (rows).

3.2.11. Silica-Fume Concrete for Bridge Decks

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Dave Whiting
Rachel Detwiler
Construction Technology Laboratories, Skokie, IL

The use of silica-fume concrete for bridge decks has become an accepted practice. This is primarily due to its favorable effects on permeability and compressive strengths. However, experience suggests that the use of silica fume in concrete contributes to high shrinkage levels that can cause deck cracking.

The primary goal of this project is to determine the effect that silica fume and other mix design parameters have on the properties of silica-fume concrete most pertinent to bridge decks. These properties include the cracking, shrinkage, diffusivity coefficient, compressive strength, and elastic modulus.

A central-composite response surface design was used to study the effect that silica fume and water-to-cement ratio have on these properties. Three independent batches of concrete were made at each of nine conditions of silica fume and water-to-cement ratio specified by the design. The design permits response surfaces to be fit for each property. The design also has the characteristic that the precision of the fitted values is independent of the direction from the center of the design.

The top figure gives the mean diffusivity coefficient, D_c, for each of the nine experimental conditions. The diffusivity coefficient measures the ability of the concrete to restrict the diffusion of chloride ions. Diffusion of chloride ions into the concrete damages the steel-bar reinforcements and ultimately destroys the concrete. The lower the D_c the better and so we see that increasing the amount of silica fume and reducing the water-to-cement ratio improves the concrete's ability to restrict the diffusion of chloride ions. The bottom figure gives a contour plot for the predicted D_c generated from fitting a second-order model to $\log(D_c)$. The plot shows that for any fixed water-to-cement ratio, increasing the amount of silica fume decreases the diffusivity coefficient. However, the decrease in D_c is much larger at lower amounts of silica fume than higher.

The investigators are currently collecting additional data. The statistical analysis of these data will provide guidance for selecting appropriate levels of silica fume for bridge decks.

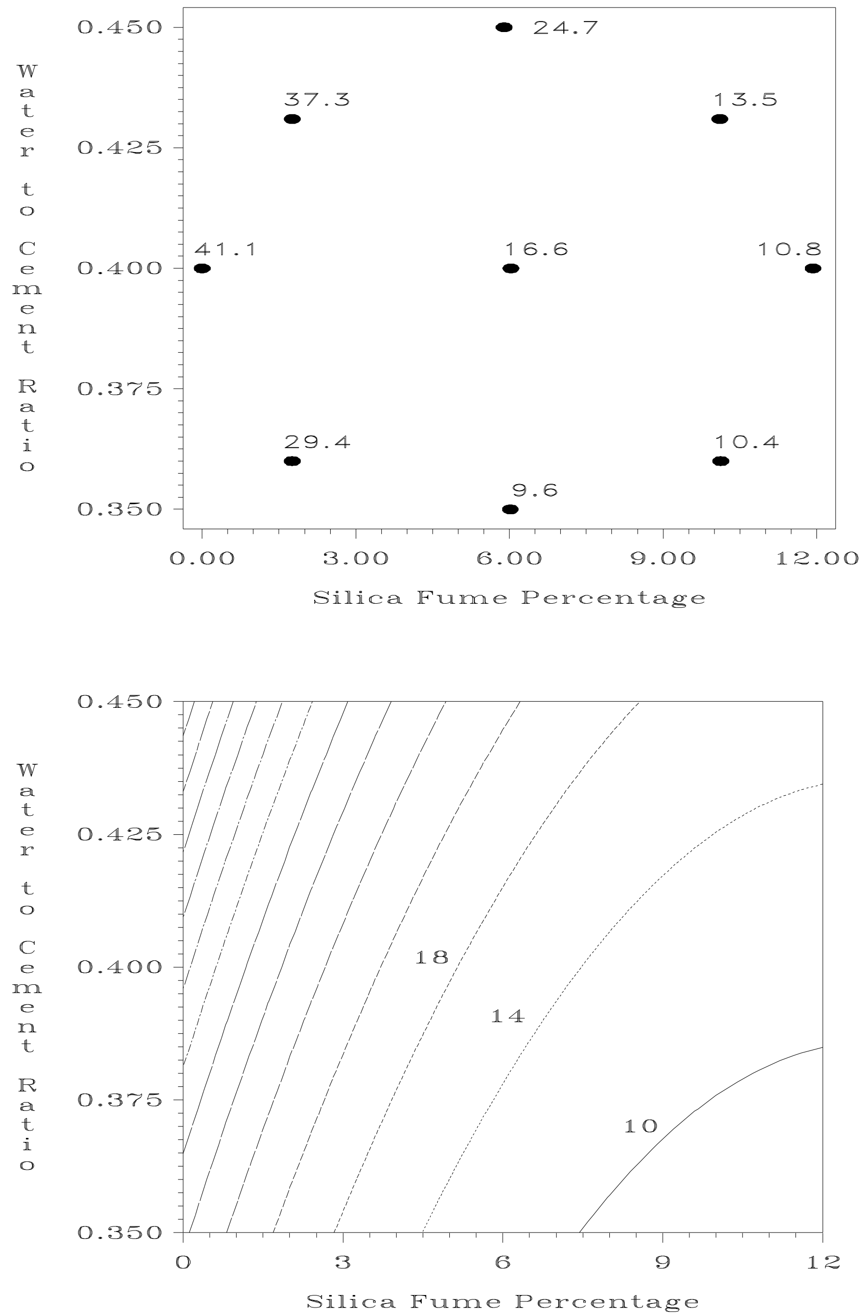

Figure 21: The top figure shows the mean diffusivity coefficient, $D_c(10^{-13})$, at each of the nine conditions of silica fume and water to cement ratio. The bottom figure is a contour plot for predicted $D_c(10^{-13})$ as a function of silica fume and water to cement ratio. Contour levels increase in steps of four units.

3.2.12. Optimizing High-Performance Concretes Using Mixture Designs

Eric S. Lagergren
James J. Filliben
Lynne B. Hare
Statistical Engineering Division, ITL

Dale P. Bentz
Ken A. Snyder
Building Materials Division, BFRL

Marcia Simon
Federal Highway Administration (FHWA)

Optimizing high-performance concrete is currently more of an art than a science. Some guidelines are available for selecting optimal conditions, but no systematic approach is used to identify these conditions. As a result, trial and error or "one-factor-at-a-time" designs are typically used to identify best mixtures. A collaboration is underway between the FHWA and NIST's Statistical Engineering and Building Materials Divisions to investigate the feasibility of using mixture design and analysis techniques for optimizing high-performance concrete. A second objective is to develop a World Wide Web service for users to design and analyze mixture experiments for optimizing concrete mixes.

In the first phase of this work, a laboratory experiment was conducted to study six mixture components: water, cement, fine and coarse aggregate, superplasticizer, and silica fume. The first four components produce concrete. The last two enhance specific properties yielding "high-performance" concrete. The properties of interest are workability, air content, strength, and chloride ion permeability. Since the proportions of the six components were constrained to a subset of the full mixture space, standard Scheffé simplex designs could not be used. Instead, a modified distance-based design was used. First, a list of candidate design points was generated including all vertices, edge centroids, constraint plane centroids, and the overall centroid. The distance criterion selects points from the candidate list to cover the experimental region in a balanced manner, maximizing the minimum Euclidean distance between points. The modification is made to ensure that the design is capable of fitting a second-order Scheffé polynomial. Additional points were included to check the adequacy of the fitted model, estimate repeatability, and check for statistical control for a total of 36 design points.

The second phase of the project is to develop a Web service for users to optimize high-performance concretes. This service will assist users in constructing mixture designs and analyzing the resulting data. Since users may have limited knowledge of statistics, the focus will be on generating informative data using good experiment design techniques and graphical methods of analysis.

The lab experiment has been completed and properties are currently being measured. Data will be analyzed by fitting an appropriate model and graphically interpreting the model via response trace plots and contour plots on 3-component simplex projections.

3.3. Statistical Inference

3.3.1. Comovement Coefficient

Stefan Leigh
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S. Perlman
National Security Agency

Andrew Rukhin
University of Maryland and Statistical Engineering Division, ITL

Time series problems encountered in numerous scientific disciplines - engineering, geophysical, biological, economic etc. - often involve the matching of two sequences for common geometric features, implying some causal or other relationship. Often the matching in the scientific literature is done numerically by the computation of a correlation coefficient. While informative to some degree, the correlation does not quantify features that the human eye readily detects as indicative of "comovement." A statistic

$$cm(u,v) \;=\; \frac{\sum \Delta u \cdot \Delta v}{(\sum(\Delta u)^2 \cdot \sum(\Delta v)^2)^{\frac{1}{2}}}$$

close to the correlation of derivatives (first differences) is proposed as a comovement coefficient. The statistic is much more relevant to comovement assay, and yet as a normalized inner product retains many of the desirable properties of the classic correlation: symmetry, translation-invariance, positive homogeneity, and so forth.

In order to estimate sampling moments/distribution of the comovement between two arbitrary time sequences, a procedure was originally proposed involving ARMA modeling of the two individual sequences, followed by innovations bootstrapping of the models in parallel, recomputing the comovement at each iteration of the bootstrap. Direct closed-form asymptotic results for the first and second moments of the comovement computed between low-order MA or AR processes have been obtained. For two AR(1) processes of the form

$$X_t = \phi_1 X_{t-1} + \epsilon_t^{(1)}, Y_t = \phi_2 Y_{t-1} + \epsilon_t^{(2)},$$

with zero-mean i.i.d. random error vectors, with arbitrary covariance structure, it can be shown that the limiting distribution is Gaussian, with mean

$$\gamma = \frac{\rho(2-\phi_1-\phi_2)\sqrt{(1+\phi_1)(1+\phi_2)}}{2(1-\phi_1\phi_2)}$$

and a variance that can be explicitly calculated. These new results, of utility and interest on their own merits, lead to modifications of the original resampling specification.

We were originally introduced to this problem during a review discussion of surface profile matching in a tribology application here at NIST.

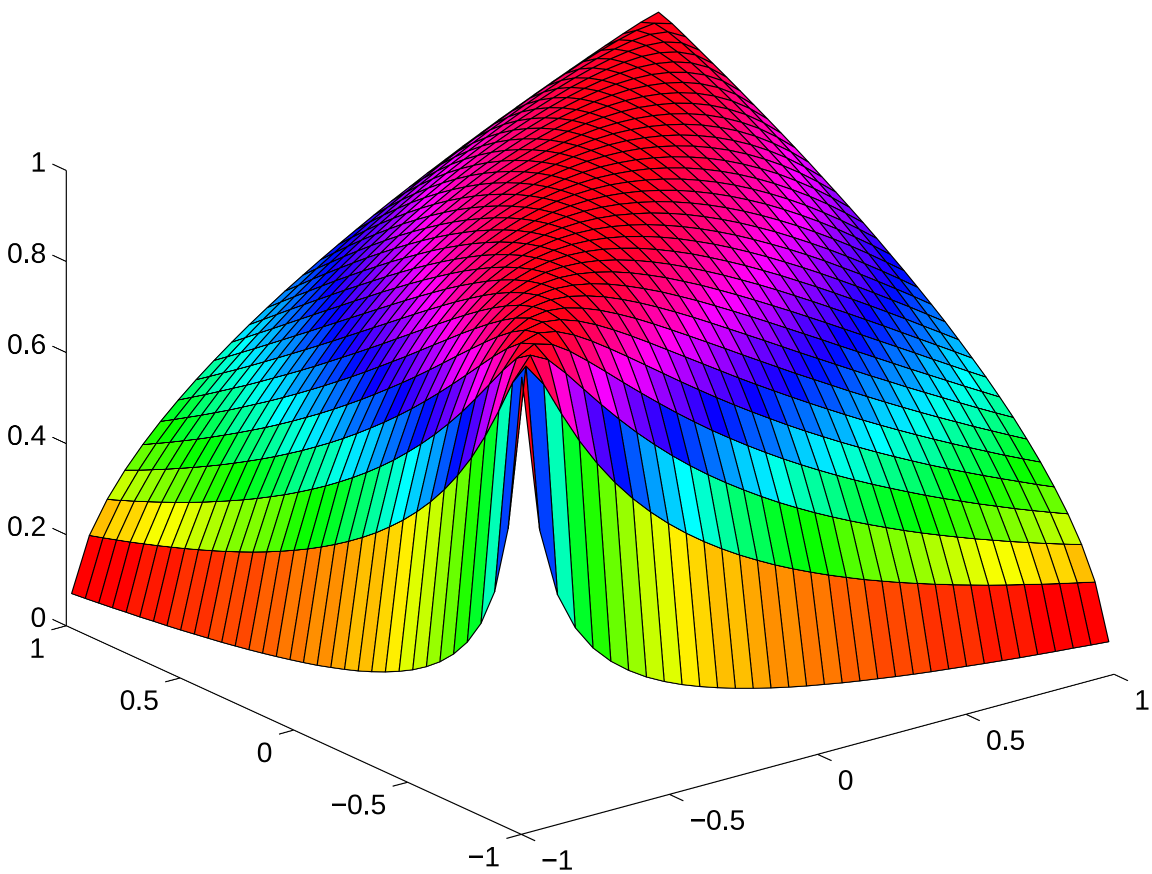

Figure 22: The limit of the sample comovement coefficient for two AR(1) processes.

3.3.2. Background Corrected Confidence Intervals For Particle Contamination Levels

Hung-kung Liu
Statistical Engineering Division, ITL

Kensei Ehara
National Research Laboratory of Metrology, Japan

In particle contamination monitoring using such instruments as laser particle counters, condensation nucleus counters, and liquid particle counters, typically an additive background noise contributes to the particle count. It frequently occurs that particle-free gas or liquid is available with which one can estimate the intensity of the instrument noise in a separate experiment.

Assume that the background noise inflated sample particle count X_s has a Poisson distribution with mean λ_s and the instrument background noise count X_n has a Poisson distribution with mean λ_n. Since background is measured in a separate experiment, we assume that X_s and X_n are independent random variables. Our parameter of interest is $\lambda = \lambda_s - \lambda_n$, which is known to be non-negative. We constructed an approximate $1 - \alpha$ confidence interval for λ

$$(X_s - X_n) + \frac{1}{2}q_{\alpha/2}^2 \pm q_{\alpha/2}\sqrt{(X_s + X_n) + \frac{1}{4}q_{\alpha/2}^2}$$

where $q_{\alpha/2}$ is the $(1-\alpha/2)$ normal quantile. This background corrected confidence interval for the contamination level is easy to compute. Compared to the uncorrected one sample Poisson confidence interval, it is shifted to correct for the background. And when there is no background noise, the proposed confidence interval degenerates to the standard one sample Poisson confidence interval.

We ran simulations to check the coverage probability for the proposed interval at $\alpha = .05$. The values of λ_s that we looked at are the integers from 1 to 30, and λ_n are all positive integers less than or equal to λ_s. For each pair of (λ_s, λ_n), we simulated 10,000 pairs of Poisson variables (X_s, X_n), and constructed 10,000 confidence intervals. The simulated coverage probability is the coverage frequency of these 10,000 confidence intervals. The following figure summarizes the simulation results.

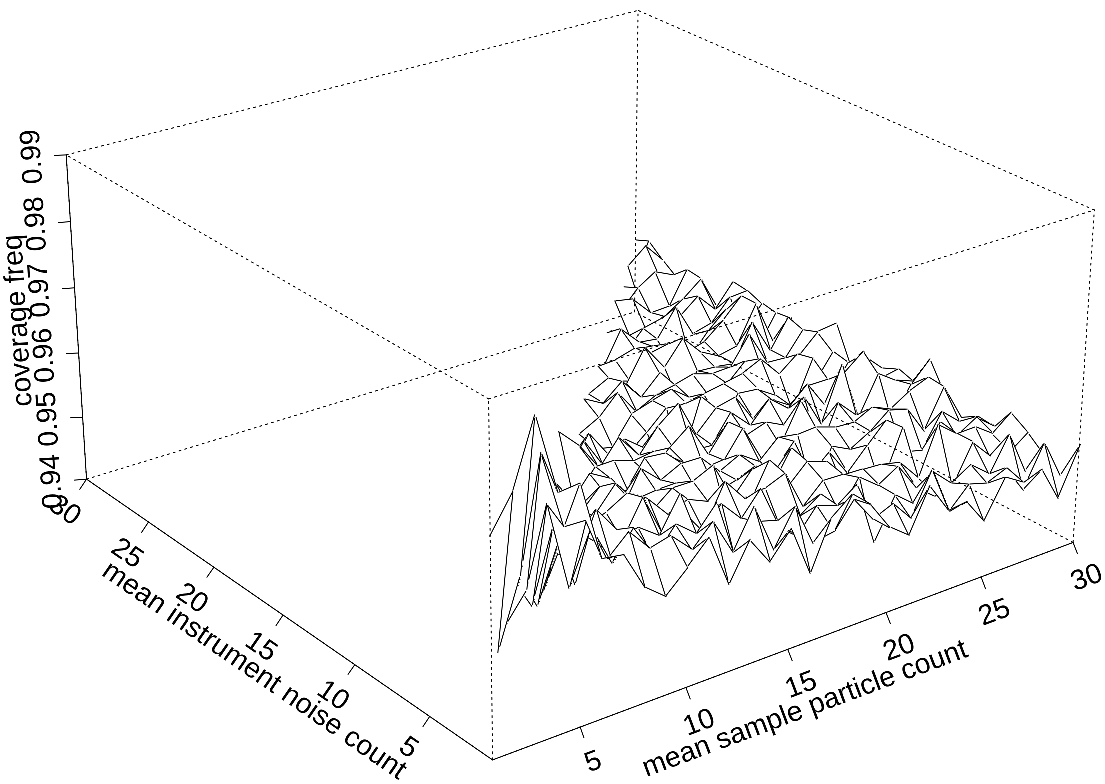

Figure 23: Simulated coverage probability for the proposed 95% confidence interval.

3.3.3. Inference on a Common Mean in an Interlaboratory Study

Mark G. Vangel
Andrew Rukhin
Bradley Biggerstaff
Stefan D. Leigh
Statistical Engineering Division, ITL

Data on a quantity measured by several laboratories often exhibits non-negligible between-laboratory variability, as well as different within-laboratory variances. Also, the number of measurements made at each laboratory can differ. A question of fundamental importance in the analysis of such data is how to best estimate a consensus mean, and what uncertainty to attach to this estimate. An estimation-equation approach due to Mandel and Paule is often used at NIST, particularly when certifying standard reference materials. However, the theoretical properties of this procedure were not well understood. Primary goals of the present research are to study the properties of this widely-used method, and to compare it with competitors, in particular to maximum-likelihood.

We have shown that the Mandel-Paule solution is equivalent to an approximate REML method, where the within-laboratory variances are estimated by the usual sample variances, instead of their restricted MLEs. Similarly, a trivial modification of Mandel-Paule can be shown to be an excellent approximation to maximum-likelihood. A very simple approximate variance for the Mandel-Paule mean estimate has been found. In numerical examples, this approximate variance agrees closely with delta-method and observed Fisher information results.

In addition, a reparametrization of the likelihood has been found which enables the entire profile-likelihood surface, in the plane of the consensus mean and between-laboratory standard deviation, to be calculated efficiently and reliably. This calculation is performed by a simple iteration which increases the likelihood with each step. By examining this surface, the MLE can be determined, along with all other stationary points. This also facilitates straightforward Bayesian computation, using a non-informative prior and numerical integration.

In the figure, the joint marginal posterior distribution for the mean and between-laboratory variance is displayed for data from an interlaboratory study in which 28 laboratories measured arsenic in NIST oyster tissue SRM 1566a. Estimates of the mean and between-laboratory standard deviations are as follows:

Method	Mean	Between-Lab. Stand. Dev.
Mandel-Paule	13.23	1.38
Modified Mandel-Paule	13.23	1.35
Maximum-Likelihood	13.22	1.36
Posterior Mode	13.23	1.34

The consensus mean posterior is also displayed, along with a 95% probability interval.

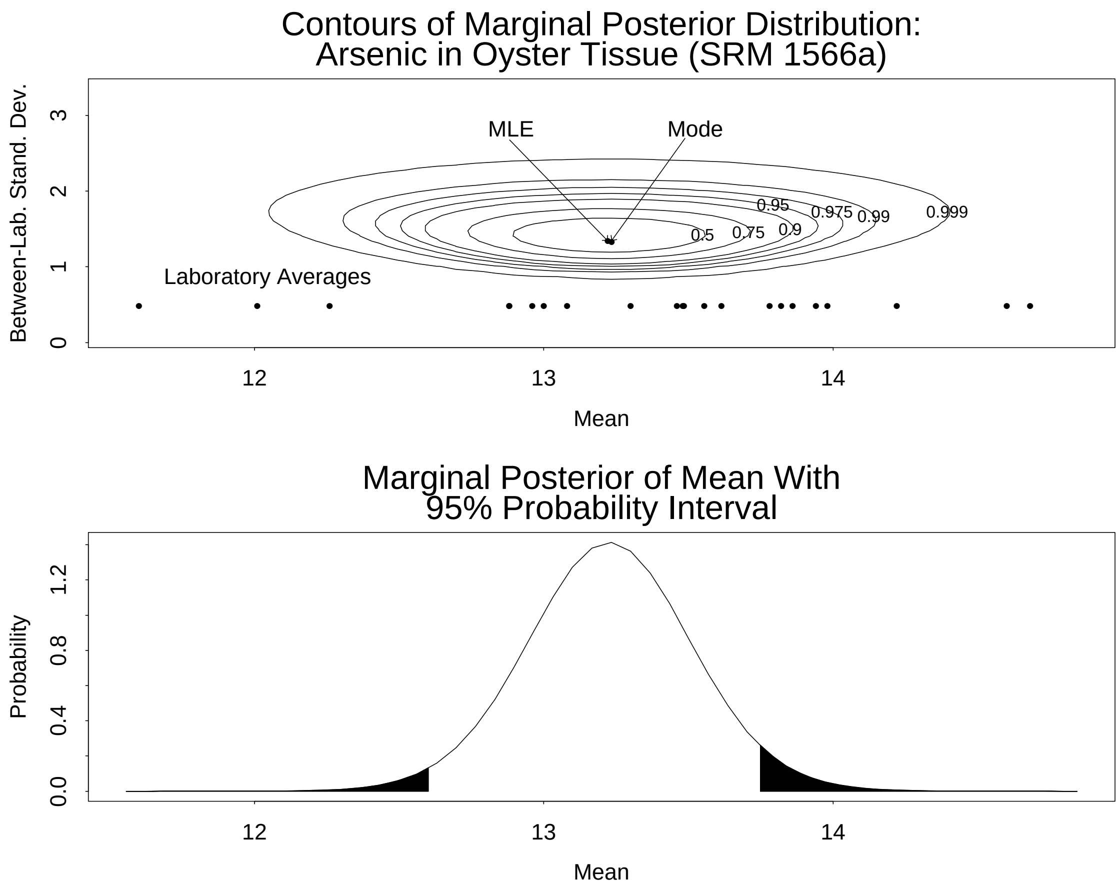

Figure 24: A Bayesian Analysis of Interlaboratory Data on Arsenic in SRM 1566a (Oyster Tissue)

3.3.4. Orthogonal Designs of User-Specified Resolution

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C. T. Liao
Colorado State University

During the initial stages of a product or a process design, engineers typically consider several factors which may influence a performance measure of interest. To understand the relative importance of each factor, it is often desirable to run one or more screening experiments. Traditionally, 2^{n-k} fractional factorial designs of resolution III, IV, or V have been used for this purpose. Sometimes, however, it is possible to obtain orthogonal designs with fewer runs than the traditional designs by searching in the class of parallel–flats designs. Because such designs need not have numbers of runs be a power of two, they may offer considerable savings in time and expense over the usual fractional factorials.

We have developed and implemented an algorithm for constructing orthogonal parallel–flats designs to meet user specifications. Specifically, we suppose an investigator can partition the full set of factorial effects into three disjoint sets:

1. Primary effects G_1: those for which unbiased estimates are required
2. Secondary effects G_2: those for which unbiased estimates are not required at this stage, but which may be nonnegligible
3. Negligible effects G_3: those believed to be negligible.

The objective is to find designs suitable for estimating all effects in G_1 based on a factorial linear model in which the effects in G_3 are assumed to be zero. Any such design is called a design of resolution (G_1, G_2). Commercial software for this problem is based on an exhaustive search for a suitable plan among single–flat designs.

Our algorithm is based on an expression for the general element of the information matrix $\mathbf{X}'\mathbf{X}$ of an arbitrary parallel–flats design, where $\mathbf{X}$ is the design matrix in the linear model $\mathbf{Y} = \mathbf{X}\beta + \epsilon$. Although the algorithm is not guaranteed to find the minimum–run design for a given problem, in nearly all of the tests conducted so far it has produced an orthogonal design with run size equal to or smaller than various published designs for estimating the same set of factorial effects.

To test the algorithm, we created several nonisomorphic sets G_1 of randomly selected primary effects with as many as 20 factors. (G_2 was taken to be the empty set for this exercise.) In each case we included all main effects, a specified number of two–factor interactions, and a specified number of three-factor interactions. Each interaction was forced to include at least one of a specified set of 1, 2, 3, or 4 "required" factors.

The table shows the success rate in finding a design smaller than the smallest possible design that could be produced by traditional search algorithms (e.g., 48 runs instead of 64; 80 or 96 runs instead of 128). In the table, n is the number of factors, x_2 is the number of two–factor interactions in G_1, x_3 is the number of three-factor interactions in G_1, r is the number of required factors, at least one of which must appear in every interaction, and the fraction $p = a/b$ shows the number a of N–run designs found in b trials. (For some problems, 100 nonisomorphic sets G_1 do not exist.)

n	x_2	x_3	r	p	N	n	x_2	x_3	r	p	N
12	20	0	2	1/1	48	16	20	0	2	20/28	48
12	20	0	3	100/100	48	16	20	0	3	6/100	48
12	20	0	4	1/100	48	16	20	0	4	0/100	48
12	18	2	2	35/100	48	16	19	1	2	7/100	48
12	18	2	3	1/100	48	16	19	1	3	1/100	48
12	18	2	4	0/100	48	16	19	1	4	1/100	48
16	16	0	2	48/49	48	18	14	0	1	1/1	48
16	16	0	3	39/100	48	18	14	0	2	50/51	48
16	16	0	4	7/100	48	18	14	0	3	24/100	48
16	15	1	2	18/100	48	18	14	0	4	4/100	48
16	15	1	3	8/100	48	20	12	0	2	41/41	48
16	15	1	4	0/100	48	20	12	0	3	46/100	48
16	14	2	2	11/100	48	20	12	0	4	0/100	48
16	14	2	3	2/100	48	18	46	0	3	3/3	80
16	14	2	4	1/100	48	18	46	0	4	26/100	96

In cases where the success rate appears to be very low (e.g., 1 out of 100), it may be the case that 48–run designs do not exist for most of the trial problems. The algorithm will find a subset of the ones that actually exist, but the number of cases for which a 48–run design exists is unknown and is generally very difficult to determine. Nevertheless, the results in the table indicate that the algorithm can be expected to be reasonably successful in finding 48–run designs of user-specified resolutions (also some 80–run and 96–run solutions).

3.3.5. Estimating Process Capability Indices for Autocorrelated Processes

Nien Fan Zhang
Statistical Engineering Division, ITL

Process capability indices (CPI) have been widely used in manufacturing industries to measure a process' performance in meeting preset specification limits. They are also used by supplier companies to demonstrate the quality of their products. Among all the capability indices, Cp and Cpk are the most widely used. In recent years there have been a lot of discussions and debates about the use of process capability indices. Interval estimation of the process capability indices was proposed. In practice, there is also a concern about the assumption of the mutual independence of the process observations. It is well known that in practice process data are often autocorrelated. This is especially true for continuous manufacturing processes such as chemical processes. When the sampling frequency is not too low, the observations are often autocorrelated. In process industries, it is common for quality personnel and process operators to use the capability indices to monitor the process performance. In this case, the variances of the sample CPI's when the data are autocorrelated are needed to construct the interval estimates of CPI.

We assume that the process is a discrete weakly stationary process. Cp and Cpk are defined in the same way as when the process observations are independent. Under the above assumption, the expectation and variance of the sample process variance were derived. It also has been shown that the covariance between the sample process mean and sample process variance is zero when the process is weakly stationary.

Approximate variances of Cp, one-sided Cpk, and Cpk have been derived in similar forms when the process observations are independent. These variances can be easily calculated based on the corresponding CPI, sample size, the process variance and autocorrelations. Thus, interval estimators of capability indices can be constructed when the process is stationary. In particular, when the process is a first order autoregressive (AR(1)) process, the approximate variances are expressed in terms of the process parameter phi, sample size, process variance and CPI. For a fixed process parameter, the attached figure shows how the variances of Cp and Cpk decrease as the sample size increases. In the figure, the curves with markers of "o", "*", and "+" correspond to the AR(1) processes with phi=0.25, 0.50 and 0.75 respectively.

Simulations have been done to find the coverage probability of k-sigma intervals of Cp and Cpk. The results show that the true Cp and Cpk lie within the interval roughly 99% of the times when k=3 and about 93% of the times when k=2.

This work was presented at the 1996 Joint Statistical Meetings.

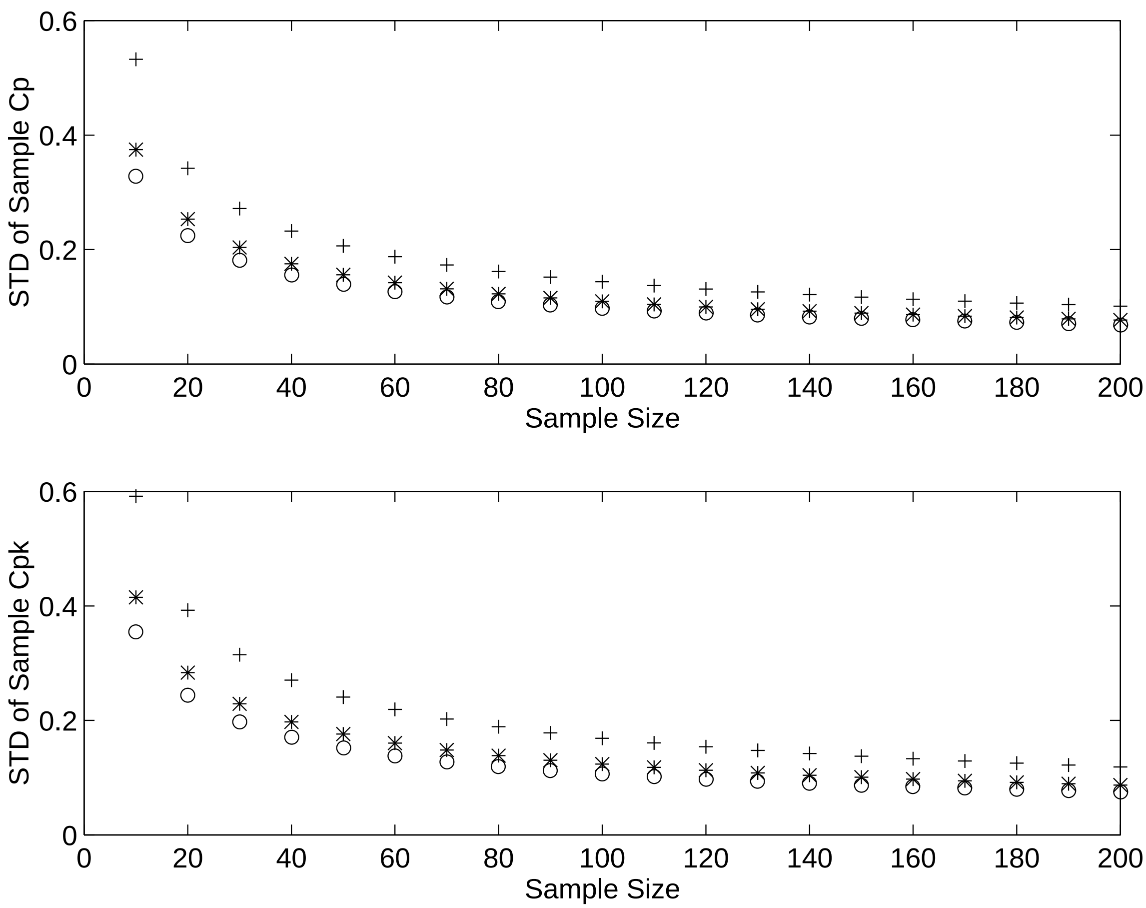

Figure 25: This figure shows how the variances of Cp and Cpk decrease as the sample size increases.

3.3.6. Long-Term Creep of Lead-Free Soldered Copper Tube Joints

Charles Hagwood
Statistical Engineering Division, ITL

Roger Clough and Donald Harne
Metallurgy Division, MSEL

Copper Development Association (CDA)

In 1993, the Copper Development Assocation (CDA) approached NIST for answers to a problem. Lead-free solders will be required in plumbing used in new building construction. Would these solders work as well as the traditional lead-containing alloys? What is the maximum safe instantaneous pressure, and the maximum allowable pressure to give a creep life of 100 years? Jointly with CDA, a set of tests was designed to provide the answers to such questions, necessary for the establishment of maximum safety plumbing codes for these new solders.

One objective of this study is to establish more precise design stresses for water system connections made with three commonly used lead-free solders. Under the auspices of the Copper Development Association, another objective is to obtain data on the long-term warm creep failure of copper tubes joined with the new lead-free solders. These data are needed in formulating new strength codes, since these solders are already being used in water systems. Our final objective is to obtain stress-lifetime data at elevated temperatures for copper tubes joined with lead-free solders and develop, with the aim of design code implementation, lifetime probability models based on these.

Anticipated outcomes are: 1) Reduced health risks and environmental degradation. 2) Improved strength and creep resistance compared to Pb/Sn Solders. 3) New tube joint codes giving cost savings to the building industry.

Millions of plumbing joints are made each year in the construction of houses, office buildings and high-rise apartments. In terms of sheer volume of solder, the building industry's use of solder is substantial compared to that of the electronics industry. Cost savings can be appreciable if the required introduction of the new lead-free solders does not require the use of more expensive soldering techniques or the use of more expensive plumbing materials by the multi-billion dollar building construction industry. Therefore these test results, which will allow the safest allowable pressures to be used, will permit optimum savings to this industry.

Failure data have been collected on solder welds at various temperatures and pressures. Most of these data sets are censored. A failure time model of the form $hours = c_0 \exp(\sigma_0 stress)$ was shown to hold and the parameters were estimated using the maximum likelihood technique under a Weibull distributional assumption. Prediction intervals have been derived.

3.3.7. A Mixed–Effects Model for the Analysis of Circular Measurements

Jack C.M. Wang
Statistical Engineering Division, ITL

C.T. Lam
Department of Industrial and Operations Engineering, University of Michigan

A circular feature in a mechanical object is one of the most basic geometric primitives. Its specification can be described easily by a center and a radius. A circular feature has several functional advantages: it has uniform strength in any direction, and its symmetry offers simplicity in assembly. However, due to imperfections introduced in manufacturing, machined parts will not be truly circular. For example, uncertainty in the positioning of the tool will cause variability in the center location; tool wear and vibration can affect the radius and the circularity of the produced features or machined parts. To estimate the geometric parameters, discrete sets of measurements are taken from machined parts. A computer controlled Coordinate Measuring Machine is commonly used for this task.

We present a statistical model for circular measurements. The proposed model captures the variation in center location of different machined parts. The radii of machined parts are assumed to be different and will be estimated from measurements. The major difference between the proposed model and the other statistical models for circular measurements studied before in the literature is that the latter models assume that the center of the true circle is fixed but unknown and hence the models do not include the between–part variation of a circular feature manufacturing process.

Under the assumption that the angular differences between measurements are known, the model is simplified to a linear model. Maximum likelihood estimates are derived for both the within and the between–part variations, as well as for the geometric characteristics. The geometric parameter estimates are compared statistically with the nominal values. A two–sided confidence interval for the between–part variability and a tolerance region which captures the population of the center of machined parts are also provided. A simple sampling scheme is obtained which minimizes the variance of the center estimate and takes into consideration the sampling cost of adding an extra machined part to the sample relative to that of taking extra measurements from machined parts. Based on this sampling scheme, statistical process control procedures can be developed to monitor the performance of the manufacturing process over time. An example on the automobile transmission gear carrier is given to illustrate the use of the results derived.

This work will appear in the May issue of *Technometrics*.

4. Conferences, Workshops and Seminars.

4.1. Quality and Uncertainty in the Measurement Laboratory

Carroll Croarkin
Keith Eberhardt
Mark Levenson
Lynne Hare
Statistical Engineering Division, ITL

Norman Belecki
Electricity Division, EEEL

Concepts of quality, measurement assurance, and uncertainty analysis for metrology were brought together in a one-day workshop presented at the ASQC Measurement Quality Division Conference in Rockville, MD on April 22-23, 1996. Belecki began the discussion by explaining the relationship between a measurement assurance approach which relies upon statistical control for assuring the quality of measurements and uncertainty analyses which are used for quantifying the goodness of measurements. He also explained the role of check standards in the measurement laboratory. Croarkin outlined the NIST policy on uncertainty and worked through a case study that illustrated the use of check standards and auxiliary experiments for estimating type A components of uncertainty. Levenson followed with a discussion and case study of type B components of uncertainty and the procedure for combining type A and B uncertainties by propagation of error techniques. Eberhardt concluded the uncertainty discussion by dealing with the difficult problem of uncertainties of measurements corrected by linear calibration. In a wrap-up session, Hare demonstrated statistical thinking as a tool for achieving quality in all scientific investigations.

4.2. Statistics Workshop, Metrology for the Americas Symposium

Carroll Croarkin
Lynne Hare
Statistical Engineering Division, ITL

Norman Belecki
Electricity Division, EEEL

Laura Alvarez-Rojas
Centro Nacional de Metrologia, Mexico

The Metrology for the Americas Symposium brought metrologists from Central and South America together with instrument manufacturers, Fluke and Hewlett Packard, and staff from NIST and the Organization of American States. The intent of the one-day statistics tutorial was to introduce the participants to statistical methods appropriate for calibration services and reference material certifications. The tutorial started with a presentation and demonstration by Hare on Statistical Thinking for Business Improvement. Measurement assurance and the role of check standards in the measurement laboratory were covered by Belecki. Error models and uncertainty were covered by Croarkin, and a Spanish translation by Alvarez of NIST Technical Note 1297, "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," was made available to the participants. Alvarez also gave an overview of her experiences as a statistician at CENAM. The tutorial ended with a demonstration of statistical software packages for solving calibration designs and analyzing data from measurement processes.

4.3. Advanced Mass Measurements

Georgia Harris
Office of Weights and Measures, Technology Services

Carroll Croarkin
Statistical Engineering Division, ITL

A workshop on mass measurements was given at NIST in June, 1996, in support of the System for Inter-American Metrology (SIM) where NIST is the lead laboratory for mass measurements. The participants, who were already skilled in mass metrology, were chosen from laboratories in Central and South America which will act as pivot laboratories for teaching and implementing advanced techniques to other laboratories within their respective regions. The workshop is part of a series that is regularly given in the USA for state and industrial laboratories, and the statistical content is substantial. It covers: theory and solution of weighing designs, propagation of uncertainties through several series of weighing designs; check standards in mass weighings, error models, computation of components of variance from check standard measurements; statistical control of the measurement process using check standards; precision of the balances; and computation of final uncertainties.

The workshop will be given again at NIST in March, 1997 for metrologists from industry and state weights and measures laboratories.

4.4. Joint Research Conference on Statistics in Quality, Industry and Technology

Eric S. Lagergren
Raghu N. Kacker
Willam F. Guthrie
Lisa M. Gill
Mark G. Vangel
Statistical Engineering Division, ITL

Timothy R. C. Read
E.I. du Pont de Nemours & Company

The 13th Quality and Productivity Research Conference and the 3rd Spring Research Conference on Statistics in Industry and Technology were held jointly at the National Institute of Standards and Technology in Gaithersburg, MD from May 29 through May 31, 1996. There were 210 statisticians and engineers that attended this first ever joint meeting. The goal of the conference was to stimulate interdisciplinary research among statisticians, engineers, and physical scientists working in quality and productivity, industrial needs, and the physical and engineering sciences. The conference featured presentations by scientists and engineers for statisticians and presentations by statisticians for scientists and engineers. Statistical issues and research approaches drawn from collaborative research were highlighted.

The program consisted of 3 plenary sessions, 26 invited talks, and 53 contributed talks. Vijay Nair opened the conference with the plenary talk "Statistics in Industry: Research Opportunities & Challenges", which outlined current research issues and models of collaboration from the author's experience. William Golomski began the second day with the plenary session "The Needs of Industry, Engineering, & Science for Statistics in the Emerging Millennium" which described critical engineering needs which call for applied statistical research. The conference closed with a plenary panel session on "Computer Models & Data Interface: Development, Validation, & Inference" led by Rob Easterling. This panel discussed statistical design and analysis issues involved when data are available from both computer models and physical tests.

There were 14 invited sessions focusing on both interdisciplinary collaborations and recent methodological advances useful for engineering applications. The invited sessions were:

1. Statistical Monitoring of Autocorrelated Processes
2. Recent Advances in Design & Analysis of Experiments
3. Statistical Thinking for Business Improvement
4. Accelerated Testing
5. Process Capability
6. Robust Design
7. Measurement of Particle Size
8. Response Surface Modeling
9. Probabilistic Methods in Image Analysis

10. Exploratory Data Analysis Tutorial
11. Integrated Circuit Burn-In Issues
12. Statistics in Information Technology
13. Statistics at SEMATECH
14. Statistics in Chemical Engineering

The fourteen contributed sessions focused on topics ranging from Experiment Design and Control Charts in Advanced SPC to Image Analysis and Stochastic Process Optimization. Each contributed session featured four 25 minute talks. Speakers from eight different countries, Canada, Croatia, India, Mexico, Singapore, South Africa, Spain, and the United States, participated in the contributed program giving the conference a true international representation.

A special effort was also made to attract students to the conference by offering a reduced registration fee and grants. Five grants covering registration and lodging were awarded. Funding for these grants was provided by the Quality & Productivity Research Conference Fund of the American Statistical Association (ASA). Twenty five students attended the conference.

The conference was sponsored by the ASA Sections on Physical & Engineering Sciences and Quality & Productivity, the Institute of Mathematical Statistics, E.I. du Pont de Nemours & Company, and the National Institute of Standards & Technology. Participants felt that this joint format for the two conferences should be repeated every several years. For more information about this conference, see
http://www.itl.nist.gov/div898/conf/jrc.html.

4.5. Construction Materials Reference Lab Statistics Course

Stefan D. Leigh
Hung-kung Liu
James J. Filliben
Mark Levenson
Statistical Engineering Division, ITL

The Construction Materials Reference Laboratory of NIST's Building Materials Division employs approximately 50 engineers, inspectors, and technicians. It provides cement, concrete and bituminous reference materials to nearly 500 voluntarily participating state and private laboratories, maintains databases of ASTM test method measurements, and engages in voluntary laboratory inspections. CMRL is increasingly being called upon to engage in a nationwide full-scale laboratory accreditation program, increasing its need for enhanced analytical capabilities. Multiple SED consulting sessions led to a suggestion that a course on data analysis geared to the needs of the CMRL might be appropriate. The result has been a series of lectures and class sessions covering a number of topics at an elementary level with content and examples deemed useful to the participants.

1. Introduction, EDA Graphics, Distributions April 28, 1995 Stefan Leigh

2. Sampling Distributions, EDA Graphics, Distributions May 12, 1995 Stefan Leigh

3. Review, Sampling Distributions, EDA Graphics, CCRL-AMRL Analyses May 25, 1995 Stefan Leigh

4. Review, Correlation, Poisson Distribution June 9, 1995 Stefan Leigh

5. Correlation June 30, 1995 Stefan Leigh

6. Regression 1 July 14, 1995 Stefan Leigh

7. Regression 1 August 11, 1995 Stefan Leigh

8. Regression 2, Gauss-Markov, Straight Line Statistics August 25, 1995 Stefan Leigh

9. Regression 3, t, Chi-Sq., F, Confidence Intervals & Inference for Straight Line September 22, 1995 Stefan Leigh

10. Hypothesis Testing November 11, 1995 Hung-kung Liu

11. Experiment Design December 1, 1995 Jim Filliben

12. Techniques for the Analysis of Designed Experiments January 19, 1996 Jim Filliben

13. The Chi-Squared Dist.: Properties, Uses, & Confidence Intervals February 2, 1996 Stefan Leigh

14. Multilinear Regression 1: Trivariate Regression March 1, 1996 Stefan Leigh

15. Multivariate Regression 2: F Dist. & Tests March 15, 1996 Stefan Leigh

16. Multivariate Regression 3: F Dist. & Tests, Leverage/Influence Statistics April 4, 1996 Stefan Leigh

17. ANOVA I April 26, 1996 Stefan Leigh

18. ANOVA II May 10, 1996 Stefan Leigh

19. Propagation of Error May 24, 1996 Stefan Leigh

20. Expressing Uncertainty of Measurement Results: The ISO and NIST Approach June 5, 1996 Mark Levenson

21. The Bootstrap June 7, 1996 Stefan Leigh

22. ANOVA III February 5, 1997 Stefan Leigh

23. ANOVA IV February 19, 1997 Stefan Leigh

23. ANOVA V March 5, 1997 Stefan Leigh

4.6. Statistics for Scientists and Engineers: A Program of Short Courses

Mark Vangel
Jim Filliben
Mark Levenson
Keith Eberhardt
Will Guthrie
Stefan Leigh
Eric Lagergren
Nien-Fan Zhang
Statistical Engineering Division, ITL

Statistical concepts and methods are indispensable to research efficiency and planning as well as the characterization of uncertainty in measurements. Hence, the Statistical Engineering Division offers a program of short courses, primarily for the NIST community. The main objective of these courses is to develop an appreciation of the meaning and usefulness of basic statistical concepts and techniques, leading at least to the ability to interpret reliably statistical analyses performed by others. In addition, sufficiently motivated scientists or engineers will be able to learn to perform their own basic statistical analyses. Each course covers some aspect of statistics, with an emphasis on applications to science and engineering problems. Together, the following courses comprise a unified program in elementary applied statistics:

- **Introduction to Statistical Concepts**
  Mark G. Vangel
  Monday 9-12, 2/26/96, 3/4/96, 3/11/96
- **Exploratory Data Analysis**
  James J. Filliben
  Monday 9-12, 4/1/96, 4/8/96, 4/15/96
- **Statistical Intervals and Uncertainty**
  Mark Levenson
  Monday 9-12, 4/29/96, 5/6/96, 5/13/96
- **Case Studies in Uncertainty Analysis**
  Keith Eberhardt
  Tuesday 9-12, 5/28/96; Monday 9-12, 6/3/96
- **Regression Models**
  William Guthrie
  Friday 9-12, 9/13/96, 9/20/96, 9/27/96
- **Analysis of Variance**
  Stefan Leigh
  Friday 9-12, 10/11/96, 10/18/96, 10/25/96

- **Design of Experiments**
  Eric Lagergren
  Friday 9-12, 11/8/96, 11/15/96, 11/22/96
- **Time Series Analysis**
  Nien-Fan Zhang
  Tuesday 9-12, 1/7/97, 1/14/97, 1/21/97

5. Special Programs

5.1. Standard Reference Materials

Carroll Croarkin
Statistical Engineering Division, ITL

The Statistical Engineering Division supports the Standard Reference Materials (SRMs) Program and the other NIST laboratories by collaborating directly with chemists and other scientists engaged in the certification of SRMs. All division staff are engaged in this activity.

Standard Reference Materials are artifacts or chemical compositions that are manufactured according to strict specifications and certified by NIST for one or more quantities of interest. SRMs represent one of the primary vehicles for disseminating measurement technology to industry.

The process of developing a new SRM can take up to five years or more and goes through several phases: 1) development and validation of a measurement method; 2) design of a prototype; 3) stability testing; 4) study of measurement error; 5) certification; and 6) uncertainty analysis. Statisticians advise on the design and analysis of experiments at all phases; develop methods for estimation for data taken by different analytical methods; reconcile interlaboratory differences; and combine all information to produce a certified value and statement of uncertainty.

In 1996, division staff collaborated on an unusually large number of SRMs, eighty or more, covering a variety of applications including: chemical (e.g., sulfur concentration in coke); health (e.g., glucose in human serum); dimensional (e.g., sinusoidal roughness); materials (e.g., diameters of polystrene spheres); environmental (e.g, lead in paint); scientific (e.g., magnification of scanning electron microscopes); and semiconductor manufacturing (e.g., resistivity of silicon wafers).

The large workload of SRMs has led the division to consider more efficient methods for handling the statistical design and analyses of SRMs. As a start, a standardized protocol for certifying gas cylinders is being developed in collaboration with chemists from CSTL. Typically, fifty or more issues of cylinders (with various gases and concentration levels) are certified per year. It is expected that once the analysis template has been coded into software, the chemists will handle the certifications with only occasional assistance from the statisticians.

If this experiment is successful, it will have three salutary effects. The time spent on SRMs within SED will decrease dramatically. The creation of software modules for other classes of SRMs with common analysis characteristics will proceed. And, NIST's long term goal of transferring measurement technology and certification capability for gas cylinders to laboratories outside of NIST will become feasible.

More information on statistical issues related to specific SRMs can be found in the body of this document.

5.2. Engineering Statistics Handbook

Carroll Croarkin, James J. Filliben, William F. Guthrie, Keith Eberhardt, Jack C.M. Wang
Statistical Engineering Division, ITL

Paul Tobias, Jack Prins, Chelli Zey
SEMATECH

Barry Hembree
AMD

Patrick Spagon
Motorola

The Statistical Engineering Division is pursuing a joint project with the Statistical Methods Group of SEMATECH to develop and publish an electronic handbook on statistical methods for scientists and engineers. The handbook will be patterned after NBS Handbook 91, *Experimental Statistics* by Mary Natrella. The updated handbook is intended to provide modern statistical and graphical techniques which are appropriate for the problems confronting the U.S. industry, particularly the semiconductor industry, and the NIST laboratories.

The handbook will also be combined with menu-driven statistical software to offer its readers a flexible, guided statistics tool kit to allow incorporation of statistical methodology into scientific and engineering work with minimum effort. The navigation through the book is structured so that the reader can get to the appropriate material via outline, engineering question, flow-chart or statistical method. The first level outline is as follows:

1. Overview
2. Looking at Data (EDA)
3. Measurement Process Characterization
4. Manufacturing Process Characterization
5. Process Modeling
6. Process Improvement

7. Process/Product Comparison
8. Process Monitoring
9. Product and System Reliability
10. Glossary of Terms and Symbols
11. Index of Engineering Questions
12. Index of Examples
13. Index of Statistical Techniques

The public domain software, Dataplot, which was developed at NIST by J. Filliben, has been extended to a menu-driven system for this purpose. In the past year, there have been several enhancements to Dataplot to allow seamless integration between the software and the handbook when viewing with a WWW browser. A unique aspect of this integration is that it allows the reader to run analyses of sample data, or his own data, directly from the handbook using a Dataplot macro.

Four prototype chapters are under development, and sections of these chapters are being made available for internal review and comment. The four prototypes will be demonstrated for the SEMATECH Advisory Council in May and featured at an invited poster session sponsored by the American Statistical Association at the Joint Statistical Meetings in Anaheim, CA, in August.

In the coming year, the prototypes will be completed; Dataplot will be extended to support the examples in the prototypes; a common structure and navigation for the handbook will be put in place; guidelines for authors will be finalized; the prototypes will be revised to a common structure; and work will begin on the other chapters.

5.3. Statistical Reference Datasets

M. Carroll Croarkin
James J. Filliben
Lisa M. Gill
William F. Guthrie
Eric S. Lagergren
Hung-Kung Liu
Mark G. Vangel
Nien-Fan Zhang
Statistical Engineering Division, ITL

Janet E. Rogers
Bert W. Rust
Mathematical & Computational Sciences Division, ITL

Phoebe Fagan
Standard Reference Data Program, TS

With the widespread use and availability of statistical software, concerns about the numerical accuracy of such software are now greater than ever. Inevitably, numerical accuracy problems can exist with some of this software despite extensive testing. Indeed, this has been a continuing cause of concern for statisticians, see e.g. Francis, Heiberger, and Velleman (American Statistician, 1975) and Eddy and Cox (Chance, 1991). Many have cited the need for an easily-accessible repository of reference datasets. To date no such collection has been available. In response to concerns of both the statistical community and industrial users, the Statistical Engineering Division in collaboration with the Mathematical & Computational Sciences Division and Standard Reference Data Program has developed a Web-based service that provides reference datasets with certified values for a variety of statistical methods. This service is called Statistical Reference Datasets (StRD).

Currently 62 datasets with certified values are provided for assessing the accuracy of software for univariate statistics, analysis of variance, linear regression, and nonlinear regression. The collection includes both generated and "real-world" data of varying levels of difficulty. Generated datasets are designed to challenge specific computations. These include the classic Wampler datasets for testing linear regression algorithms and the Simon & Lesage datasets for testing analysis of variance algorithms. Real-world data include challenging datasets such as the Longley data for linear regression, and more benign datasets such as the Daniel & Wood data for nonlinear regression.

Certified results for linear procedures were obtained using extended precision software to code simple algorithms for each type of computation. Carrying 500 digits through all of the computations allowed calculation of output unaffected by floating point representation errors. Certified values for nonlinear regression are the "best-available" solutions, obtained using 64-bit precision and confirmed by at least two different algorithms and software packages using analytic derivatives.

In the coming year, the team will be publicizing the StRD web service. A special contributed paper session, "Statistical Reference Datasets (StRD) for Assessing the Numerical Accuracy of Statistical Software," will be presented at the 1997 Joint Statistical Meetings in Anaheim, CA.

5.4. MIL-HDBK-17: Composite Materials Handbook

Mark Vangel
Statistical Engineering Division, ITL

Mark Vangel is Chairman of the Statistics Working Group of Mil-Handbook-17, which develops and publishes statistical methods for composite materials. These materials, which can have exceptionally high ratios of strength and stiffness to weight, are of growing importance, particularly in the aerospace industry. However, strength properties of composite materials typically exhibit considerable variability, due to the brittleness of most fibers and many matrices, and due to processing. Statistical methods (specifically methods for tolerance limits, mixed model analysis, and quality control) are thus important to the use of these materials. Mil-Handbook-17 is an evoloving document which is intended to be used as a primary reference, both for data and for guidelines on data analysis, by composites engineers, by the Department of Defense, and by regulatory agencies.

6. Staff Publications and Professional Activities

6.1. Publications

6.1.1. Publications in Print

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30. C.M. Wang, (with J.R. Juroshek, G.P. McCabe), Statistical analysis of network analyzer measurements, *IEEE Trans. Instrumentation and Measurement*, submitted.
31. C.M. Wang, (with P.A. Williams, A.H. Rose), Rotating–polarizer polarimeter for accurate retardance measurement, *Applied Optics*, submitted.
32. C.M. Wang, (with K.B. Rochford), Accurate interferometric retardance measurements, *Applied Optics*, submitted.
33. C.M. Wang, (with A.H. Rose, S.M. Etzel), Verdet constant dispersion in annealed optical fiber current sensors, *Journal of Lightwave Technology*, submitted.
34. G.L. Yang, The Kaplan-Meier estimator, *Encyclopedia of Statistical Science*, Wiley, 1997, to appear.
35. G.L. Yang, Le Cam's procedure and sodium channel experiments, *Research Papers in Statistics and Probability, a Festschrift for Le Cam*, 1997, Springer-Verlag, to appear.
36. G.L. Yang, Markov Chains and Nerve Impulses: An Interface between Statistics and Neurophysiology , *A festschrift for S. Kotz.*, 1997, Wiley, to appear.
37. G.L. Yang, (with D. Pollard, E. Torgersen), Co-editor, *Research Papers in Probability and Statistics: A festschrift for Lucien Le Cam,*,1997, pp. 470, Springer, to appear.
38. G.L. Yang, Comparing censoring and random truncation via nonparametric estimation of a distribution function, *Statistical Challenges in Modern Astronomy, II*, Springer, E.D. Feigelson and G.J. Babu, eds, to appear.
39. G.L. Yang, (with S. He), The strong law under random truncation, submitted.

40. G.L. Yang, (with S. He), Estimation of the truncation probability in the random truncation model, *Annals of Statistics* under revision.
41. N.F. Zhang, Detection Capability of Residual Control Chart for Stationary Processes, *Journal of Applied Statistics*, to appear in 24(2), 1997.
42. N.F. Zhang, Autocorrelation Analysis of Some Linear Transfer Function Models and Its Applications in the Dynamic Process Systems, to appear in Lectures in Applied Mathematics: Proceedings of 1996 AMS-SIAM Summer Seminar.
43. N.F. Zhang, Estimating Process Capability Indices for Autocorrelated Processes, submitted for publication, 1996.
44. N.F. Zhang, A Statistical Control Chart for Stationary Process Data, submitted for publication, 1996.

6.1.5. Working Papers

1. K.J. Coakley, C. Hagwood, H.K. Liu and D.S. Simons, Detection and Quantification of Isotopic Inhomogeneity.
2. K.J. Coakley, Optimal Design of Neutron Lifetime Experiment.
3. K.R. Eberhardt, (with B. Belzer and J.R. Ehrstein), CN–1364 NIST/VLSI thin film standards: final report.
4. L.M. Gill, J.J. Filliben, S. Jahanmir, L. Ives, Effects of Grinding on Strength of Reaction Bonded Silicon Nitride.
5. L.M. Gill, J.J. Filliben, S. Jahanmir, L. Ives, Effects of Grinding on Strength of Sintered Reaction Bonded Silicon Nitride.
6. L.M. Gill, J.J. Filliben, S. Jahanmir, L. Ives, Effects of Grinding on Strength of Sintered Silicon Nitride.
7. W.F. Guthrie (with N.J. Carino and G.M. Mullings), Evaluation of ASTM Standard Consolidation Requirements for Preparing High-Strength Concrete *Proc. of the ACI International Conf. on High-Performance Concrete*, Kuala Lumpur, Malaysia, December 2-5, 1997.
8. S.D. Leigh, S. Perlman, A.L. Rukhin, A Comovement Coefficient for Time Sequences.
9. H.K. Liu, (with J.T. Hwang) Testing for nonmodal error using high-dimensional empirical linear prediction.
10. M.G. Vangel, A User's Guide to RECIPE: A FORTRAN Program for Determining Regression Basis Values (version 1.0), 1995.
11. M.G. Vangel, (with D.G.M. Anderson), Richardson's Algorithm and the Approximate Solution of Singular and Inconsistent Matrix Equations.
12. G.L. Yang, (with N.F. Zhang), A modified process capability index.
13. N. F. Zhang, (with M.T. Postek, R.D. Larrabee, A. E. Vladar, W.J. Keery and S.N. Jones), A Statisitcal Measure for the Sharpness of SEM Images.
14. N. F. Zhang, A Multivariate Exponentially Weighted Moving Average Control Chart for Stationary Processes.

6.1.6. Acknowledgements in Publications

1. K.J. Coakley acknowledged in: Observation of Bose-Einstein Condensation in a Dilute Atomic Vapor, M.H. Anderson, J.R. Ensher, M.R. Matthews,C.E. Wieman, E.A. Cornell, *Science*, 269, 198(1995).
2. K.R. Eberhardt, J.J. Filliben, M.S. Levenson acknowledged in: J.E. Norris, G.A. Klouda, and E.M. Eijgenhuijsen, Ozone Calibration at NIST, presentation at National Conference of Standards Laboratories Workshop and Symposium, July, 1996.
3. K.R. Eberhardt, M. Vangel, and A. Rukhin acknowledged in: R.N. Kacker, N.F. Zhang and R.C. Hagwood, Real time control of a measurement process, *Metrologia*, 33 (5), 1996, pp. 433–445.
4. W.F. Guthrie acknowledged in: B.W. Mangum, E.R. Pfeiffer, G.F. Strouse, J. Valencia-Rodriguez, J.H. Lin, T.I. Yeh, P. Marcarino, R. Dematteis, Y. Liu, Q. Zhao, A.T. Ince, F. Cakrioglu, H.G. Nubbemeyer, H.-J. Jung, Comparisons of Some NIST Fixed-Point Cells with Similar Cells of Other Standards Laboratories, *Metrologia*, 1996, 33, pp. 215-225.
5. E.S. Lagergren acknowledged in: R.R. Zarr, Room-Temperature Thermal Conductivity of Expanded Polystyrene Board for a Standard Reference Material, NISTIR 5838.
6. S.D. Leigh acknowledged in: Extreme Wind Distribution Tails: A Peaks over Threshold" Approach, E. Simiu, N.A. Heckert, *Journal of Structural Engineering*, 122(5), 1996, p. 546.
7. M.S. Levenson acknowledged in: S.M. Stigler, Statistics and the Question of Standards, *Journal of Research of the National Institute of Standards and Technology*, Vol 101, 1996, pp. 779-789.
8. W.S. Liggett acknowledged in: D.S. Pallett, J.G. Fiscus, 1996 Preliminary Broadcast News Benchmark Tests, *Proceedings of the DARPA 1997 Speech Recognition Workshop*, to appear.
9. D.F. Vecchia acknowledged in: T.J. Bruno, K.H. Wertz, M. Caciari, Kovats retention indices for halocarbons on a hexafluoropropylene epoxide–modified graphitized carbon black, *Analytical Chemistry* 68(8) (1996).

6.2. Talks

6.2.1. Technical Talks

1. K.J Coakley, Optimal Design of Neutron Lifetime Experiment, Technical Collaboration Meeting at Harvard University, October, 1996.
2. K.J Coakley, Chaotic Behavior of Marginally Trapped Neutrons, Technical Collaboration Meeting at Harvard University, October, 1996.
3. C. Croarkin, ISO and NIST Uncertainty Policies, 1996 Measurement Quality Conference, Rockville, MD, Apr. 23, 1996.
4. C. Croarkin, Type A Uncertainities - A Case Study, 1996 Measurement Quality Conference, Rockville, MD, Apr. 23, 1996.

5. C. Croarkin, Calibration Designs, Workshop on Statistics in Metrology, Guanajuato, Mexico, Aug. 6, 1996.
6. C. Croarkin, Using Check Standards to Determine Uncertainty, Workshop on Statistics in Metrology, Guanajuato, Mexico, Aug. 8, 1996.
7. K.R. Eberhardt, Statistical uncertainty analysis of standard reference materials, Pittsburgh Conference on Chemistry and Applied Spectroscopy, Chicago, IL, March 6, 1996.
8. L.M. Gill, Design and Analysis of an Experiment to Characterize the Effect of Grinding Parameters on the Strength of Sintered Reaction Bonded Silicon Nitride, NIST Ceramic Machining Consortium, 8th Program Review Meeting, March 28, 1996.
9. L.M. Gill, Reference Materials: An Overview to Understand the Needs and Uses, CENAM, Queretaro, Mexico, October 28, 1996.
10. L.M. Gill, Reference Materials: Design Issues in the Certification of Reference Materials, CENAM, Queretaro, Mexico, October 29, 1996.
11. L.B. Hare, A Case Study in the Reduction of Variation Through Graphics and Statistical Thinking, George Mason University, Fairfax, VA, Apr. 19, 1996.
12. L.B. Hare, Statistical Thinking for Business Improvement, Measurement Quality Conference, Rockville, MD, Apr. 23, 1996.
13. L.B. Hare, Statistical Thinking and the Control of Key Food Processing Factors, American Society for Quality Control, Annual Quality Congress, Chicago, IL, May 13, 1996
14. L.B. Hare, Statistical Thinking for Work Process Improvement, Workshop on Statistics in Metrology, Guanajuato, Mexice, August 5-9, 1996.
15. L.B. Hare, Statistical Thinking and the Control of Key Food Processing Factors, American Society for Quality Control, Salisbury, MD, October 17, 1996.
16. L.B. Hare, (with G. Britz, D. Emerling, R. Hoerl, J. Shade) How to Implement Statistical Thinking Effectively, American Society for Quality Control and American Statistical Association Fall Technical Conference, Scottsdale, AZ, October 25, 1996.
17. L.B. Hare, Design and Analysis of Mixture Experiments, Rutgers University Advanced Design of Experiments Course, New Brunswick, NJ, December 5, 1996.
18. E.S. Lagergren, Statistical Reference Datasets, Town Meeting on On-line Delivery of NIST Standard Reference Data, NIST, Gaithersburg, MD, November 19, 1996.
19. M.S. Levenson, Examples in Statistical Image Processing, NIST, Gaithersburg, MD, February, 1996.
20. M.S. Levenson, Type B Uncertainties and More—A Case Study, Measurement Quality Conference, Rockville, MD, April, 1996.
21. M.S. Levenson, Adaptive Smoothing of Images with Local Weighted Regression, Joint Research Conference, NIST, Gaithersburg, MD, May, 1996.
22. M.S. Levenson, An Overview of the ISO/NIST Uncertainty Policy, NIST, Gaithersburg, MD, June, 1996.
23. M.S. Levenson, Removing Quantization Noise in Images Using Wavelets, Joint Statistical Meetings, Chicago, IL, August, 1996.
24. M.S. Levenson, An Overview of the ISO/NIST Uncertainty Policy, Laser Measure-

ment Short Course, Boulder, CO, August, 1996.

25. M.S. Levenson, Statistical Aspects of Certifying Reference Materials, Centro Nacional de Metrología, Querétero, México, October, 1996.
26. W.S. Liggett, (with K.W. Moon and C.A. Handwerker) An Experimental Method for Refinement of Solderability Measurement, 1996 Fall Technical Conference, Scottsdale, AZ, October 24, 1996.
27. W.S. Liggett, (with R.A. Fletcher) Experimental Characterization of Optical Particle Counters, 1996 Joint Research Conference on Industrial Statistics, Gaithersburg, MD, May 30, 1996.
28. W.S. Liggett, Geostatistics for Engineered Surfaces, Applications of Statistics: What's Hot/What's Next, Troy, NY, April 27, 1996
29. H.K. Liu, Multivariate data analysis using empirical linear models, the 1996 Joint Statistical Meetings, August 6, 1996, Chicago, IL.
30. H.K. Liu, Background corrected statistical confidence intervals for particle contamination levels, 13th International Symposium on Contamination Control, September 17, 1996, the Hague, the Netherlands.
31. H.K. Liu, High-dimensional empirical linear prediction, Euroconference–Advanced Mathematical Tools in Metrology III, September 28, 1996, Berlin, Germany.
32. A.L. Rukhin, Estimation of a Projection as a Neyman-Scott Problem, Sixth Eugene Lucacs Symposium, Bowling Green University, Bowling Green, Ohio, March 29, 1996,
33. A.L. Rukhin, Adaptive Testing of Multiple Hypotheses for Stochastic Processes, AMS-IMS-SIAM Joint Summer Research Conference on Adaptive Selection of Models and Statistical Procedures, Mount Holyoke College, S. Hadley, MA, June 27, 1996.
34. A.L. Rukhin, Bayes Estimation in the Change-Point Problem, Indiana University-Purdue University at Indianapolis, Indianapolis IN, October 16, 1996.
35. A.L. Rukhin, Adaptive Multiple Decisions for Random Processes and Fields, Workshop on Stochastic Analysis, Michigan State University, E. Lansing MI, October 19, 1996.
36. M.G. Vangel, Inference on a Common Mean in an Interlaboratory Study, Statistical Engineering Division Colloquium, NIST, Gaithersburg, MD, January 24, 1996.
37. M.G. Vangel, Applications of Statistical Tolerance Limits in Aircraft Design, Reliability Center, University of Maryland, College Park, MD, March 7, 1996.
38. M.G. Vangel, New Results for the Analysis of a Series of Similar Experiments, Joint Conference on Statistics in Research, Industry, and Technology, Gaithersburg, MD, May 30, 1996.
39. A.L. Rukhin and M.G. Vangel,Estimation of a Common Mean and Weighted Means Statistics, Joint Conference on Statistics in Research, Industry, and Technology, Gaithersburg, MD, May 30, 1996.
40. M.G. Vangel, One-Sided Mixed-Model Tolerance Limits, Joint Statistical Meetings, Chicago, IL, August 5, 1996.
41. A.L. Rukhin and M.G. Vangel , Inference on a Common Mean in Interlaboratory Studies, Joint Statistical Meetings, Chicago, IL, August 6, 1996.
42. M.G. Vangel and A.L. Rukhin, Combining Information in Interlaboratory Studies,

NIST Colloquium, Boluder, CO, September 3, 1996.

43. G.L. Yang, The probability of truncation and the strong law under random truncation, The Joint Statistical Meeting, Chicago, August 10, 1996.
44. G.L. Yang, Estimation of the truncation probability in the random truncation model, Colloquium, Johns Hopkins, October 11, 1996.
45. N.F. Zhang, A New Algorithm for the Measurement of Pitch in Metrology Instruments, SPIE's 1996 International Symposium on Microlithography, Santa Clara, CA, March, 1996.
46. N.F. Zhang, Some Issues in Applications of Process Capability Indices, Joint Research Conference on Statistics in Quality, Industry, and Technology, Gaithersburg, MD, May, 1996.
47. N.F. Zhang, Autocorrelation Analysis of Some Linear Transfer Function Models and Its Applications in the Dynamic Process Systems, 1996 AMS-SIAM Summer Seminar, Williamsburg, VA, June 1996.
48. N.F. Zhang, Estimating Process Capability Indices for Autocorrelated Processes, 1996 Joint Statistical Meetings, Chicago, IL, August 1996.

6.2.2. General Interest Talks

1. K.J Coakley, Nonequilibrium Kinetics of Neutral Atoms in a Harmonic Potential, University of Chile, Physics Department, March, 1996.
2. K.J Coakley, A Bootstrap Method for Nonlinear Filtering of EM-ML Reconstructions of PET Images, Catholic University, Santiago, Chile, Mathematics Department and University of Chile, Applied Mathematics Department, March, 1996.
3. C. Croarkin, History of NIST/SEMATECH Handbook to SEMATECH Advisory Council, San Antonio, TX, Apr. 24,1996.

6.2.3. Workshops for Industry

1. C. Croarkin, M. Levenson, K. Eberhardt (with N. Belecki), Workshop on Quality and Uncertainty for Measurement Laboratories at the 1996 Measurement Quality Conference, Rockville, MD, Apr. 23, 1996.
2. C. Croarkin (with G. Harris), Workshop on Advanced Mass Measurements, NIST, Gaithersburg, Mar. 10-12, 1997.
3. K.R. Eberhardt (with E.W. de Leer, R.L. Watters), Uncertainty Calculations in Chemical Measurements, Short Course 414, The Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 18, 1997.

6.2.4. Lecture Series

1. K.R. Eberhardt, Uncertainty Analysis Case Studies, part of the series on Statistics for Scientists and Engineers, NIST, Gaithersburg, MD, June 10 and 17, 1996.

2. L.M. Gill, Reference Materials: An Overview to Understand the Needs and Uses, CENAM, Queretaro, Mexico, October 28, 1996.
3. L.M. Gill, Reference Materials: Design Issues in the Certification of Reference Materials, CENAM, Queretaro, Mexico, October 29, 1996.
4. E.S. Lagergren, Design of Experiments, Statistics for Scientists & Engineers Series, Gaithersburg, MD, November 8, 15, and 22, 1996.
5. M.S. Levenson, Intervals and Uncertainty Analysis, NIST, Gaithersburg, MD, April and May, 1996.
6. M.G. Vangel, Statistical Methods for Composite Material Basis Properties, a short-course presented at the Composite Materials Handbook meeting, Schaumburg, IL, September 12, 1996.

6.3. Professional Society Activities

6.3.1. NIST Committee Activities

1. K.J Coakley, served on Boulder Editorial Review Board.
2. K.J Coakley, Member, EEEL CALCOM committee for SRM 2538 (Coplaner Waveguide).
3. C. Croarkin, Member, NIST Standards Advisory Committee.
4. C. Croarkin, Member, EEEL CALCOM Committee on Resistivity.
5. C.M. Wang, Member, EEEL CALCOM Committee on Optical Fiber Chromatic Dispersion Standard.
6. C.M. Wang, Member, EEEL CALCOM Committee on Optical Retardance Standard.

6.3.2. Standards Committee Memberships

1. C. Croarkin, Vice-Chair, US TAG to ISO TC-69 on Statistical Methods.
2. C. Croarkin, Chair, ANSI ASC Statistics Subcommittee.
3. C. Croarkin, Member, ASTM E-11 Subcommittee on Quality and Statistics.
4. S.D. Leigh, Member, ASTM/ISR Reference Soil & Testing Program.

6.3.3. Other Professional Society Activities

1. K.R. Eberhardt, Chaired contributed paper session on Issues in Sampling, Joint Research Conference on Statistics in Quality, Industry and Technology, NIST, Gaithersburg, MD, May 29, 1996.
2. L.M. Gill, Secretary / Treasurer of the ASA Quality and Productivity Research Conference Steering Committee, 3 year term.
3. L.B. Hare, Chair, Section on Quality and Productivity, American Statistical Association, 1997.

4. L.B. Hare, Awards Committee Chair, Statistics Division, American Society for Quality Control, 1995-1997.
5. L.B. Hare, Committee Member, Statistical Partners in Academe, Industry and Government, American Statistical Association, 1996-1997.
6. E.S. Lagergren, R.K. Kacker, L.M. Gill, W.F. Guthrie, Organizing Committee, ASA/IMS Joint Research Conference, NIST, Gaithersburg, MD, May 29-31, 1996.
7. E.S. Lagergren, Steering Committee Member, ASA Quality and Productivity Research Conference, 1995–1997.
8. E.S. Lagergren, Program Chair-Elect, ASA Quality and Productivity Section, 1997–1998.
9. W.S. Liggett, Editor, American Society for Quality Control, Statistics Division, How-To Series, 1995–.
10. H.K. Liu, Chaired the Session on Process Monitoring & Control, Joint Research Conference on Statistics in Quality, Industry, and Technology, NIST, Gaithersburg, MD, May 29, 1996.
11. A.L. Rukhin, IMS representative to AAAS, 1996-1998.
12. G.L. Yang, Council member, Institute of Mathematical Statistics, 1994–1997.
13. G.L. Yang, Council member, Bernoulli Society, 1995–1999.
14. G.L. Yang, Program Chair, 1996 Annual Meeting of the Institute of Mathematical Statistics.

6.4. Professional Journals

6.4.1. Editorships

1. L.B. Hare, Management Committee, Technometrics.
2. W.S. Liggett, Associate Editor, Environmental and Ecological Statistics.
3. A.L. Rukhin, Associate Editor, Statistics&Probability Letters.
4. A.L. Rukhin, Associate Editor, Statistics&Decisions.
5. A.L. Rukhin, Associate Editor, Mathematical Methods of Statistics.
6. A.L. Rukhin, Associate Editor, Applicationes Mathematicae.
7. G.L. Yang, Associate Editor, Journal of Statistical Planning and Inference.

6.4.2. Refereeing

1. K.J. Coakley, IEEE Transactions on Medical Imaging, Journal of the American Statistical Association, Computational Statistics and Data Analysis.
2. K.R. Eberhardt, Technometrics, Journal of Quality Technology, Annals of Statistics.
3. E.S. Lagergren, Journal of Quality Technology.
4. M.S. Levenson, IEEE Transactions on Medical Imaging.
5. W.S. Liggett, Statistics and Probability Letters.
6. H.K. Liu, Annals of Statistics, Academia Sinica.

7. A.L. Rukhin, Annals of Statistics, Statistical Papers, Communications in Statistics.
8. M.G. Vangel, Statistica Sinica, Statistics and Probability Letters, Journal of the American Statistical Association, IEEE Journal of Rehabilitation Engineering, SAMPE Journal of Advanced Materials.
9. C.M. Wang, Communications in Statistics.
10. N.F. Zhang, Applied Radiation and Isotopes, IEEE Transactions on Automatic Control, Computational Statistics and Data Analysis.

6.5. Proposal Reviewing

1. K.R. Eberhardt, NIST Advanced Technology Program.
2. N.F. Zhang, NIST Advanced Technology Program, DARPA and NIST (TREC Project).

6.6. Honors

1. W.S. Liggett (with S.R. Low, D.J. Pitchure, T.V. Vorburger, J.F. Song), Edward Bennett Rosa Award, December, 1996

6.7. Trips Sponsored by Others and Site Visits

1. L. Hare and C. Croarkin (with N. Belecki), Presented several talks related to metrology as part of the series, Verano de Estadistica Industrial, sponsored by the Centro Investigacion de Mathematica de Mexico, Guanajuato, Aug. 5-9, 1996.
2. L.M. Gill, and M. Levenson, Visit to CENAM, Queretaro, Mexico, October 28 - November 1, 1996.
3. E.S. Lagergren, Meet with Project Review Panel, Construction Technology Laboratories, Inc., Skokie, IL, April 22, 1996.
4. M.S. Levenson and L.M. Gill, Visit to Centro Nacional de Metrología, Querétero, México, October 28 – November 1, 1996.
5. H.K. Liu, Euroconference–Advanced Mathematical Tools in Metrology III, September 25-28, 1996, Berlin, Germany.
6. M.G. Vangel, Mil-Handbook-17 (Composite Materials Handbook) Coordination Group Meeting, Santa Fe, NM March 1996 (Trip sponsored by the Army Research Laboratory, Materials Directorate).
7. M.G. Vangel, Mil-Handbook-17 (Composite Materials Handbook) Coordination Group Meeting, Schaumburg, IL, September 1996 (Trip sponsored by the Army Research Laboratory, Materials Directorate).

6.8. Training & Educational Self-Development

1. K.R. Eberhardt, EEO for Supervisors and Managers, NIST, Gaithersburg, MD, May 7, 1996.
2. K.R. Eberhardt, Classification Training for Supervisors, NIST, Gaithersburg, MD, May 28, 1996.
3. K.R. Eberhardt, Demonstration Project Performance Management System, NIST, Gaithersburg, MD, July 22, 1996.
4. K.R. Eberhardt, Health and Safety Responsibilities, NIST, Gaithersburg, MD, July 24, 1996.
5. K.R. Eberhardt, Position Sensitivity and Security Investigations, NIST, Gaithersburg, MD, September 16, 1996.
6. K.R. Eberhardt, Conflict Resolution, NIST, Gaithersburg, MD, October 23, 1996.
7. K.R. Eberhardt, Diversity in the Workplace, NIST, Gaithersburg, MD, November 14, 1996.
8. K.R. Eberhardt, Distributed Computing and Information Services, NIST, Gaithersburg, MD, December 2, 1996.
9. K.R. Eberhardt, EEO for Managers and Supervisors, NIST, Gaithersburg, MD, December 4, 1996.
10. K.R. Eberhardt, NIST Employee Assistance Program, NIST, Gaithersburg, MD, January 15, 1997.
11. L.M. Gill, You Can Make a Difference in the Workplace and in Your Life, Federally Employed Women, DC Metro Regional Training Program, Bethesda, MD, March 7-8, 1996.
12. L.M. Gill, First Things First: Covey Principles, Washington, D.C., Nov. 13, 1996.
13. L.M. Gill, Financial Planning Workshop: Planning for Retirement, NIST, Gaithersburg, November 18, 1996.
14. E.S. Lagergren, Making Meetings Work, NIST, Gaithersburg, MD, April 2, 1996.
15. E.S. Lagergren, Effective Team Building, NIST, Gaithersburg, MD, May 14, 1996.
16. E.S. Lagergren, Technical Writing, NIST, Gaithersburg, MD, May 21-22, 1996.
17. E.S. Lagergren, First Things First, Washington, DC, July 9, 1996.
18. E.S. Lagergren, Seven Habits of Highly Effective People, Bethesda, MD, December 17-19, 1996.

6.9. Special Assignments

1. L.M. Gill, SRM Team Leader.
2. L.M. Gill, Local Arrangements Chair, Organizing Committee, Joint Research Conference in Statistics in Quality, Industry, and Technology.
3. L.M. Gill, Assistant EEO Coordinator for ITL.
4. E.S. Lagergren, M.C. Croarkin, J.J. Filliben, L.M. Gill, W.F. Guthrie, H.K. Liu, M.G. Vangel, N.F. Zhang (with P. Fagan, J.E. Rogers, B.W. Rust), Statistical Reference Datasets Team, 1996.
5. E.S. Lagergren, SRM Team Leader, 1996.

6. S.D. Leigh, NRC postdoctoral associateship coordinator for SED.
7. H.K. Liu, Coordinator, SED Seminar Series, Gaithersburg, MD.
8. M.G. Vangel, Chairman, Statistics Working Group, Mil-Handbook-17 (Composite Materials Handbook) Coordination Group.
9. M.G. Vangel, Program Committee Member, Army Conference on Applied Statistics.